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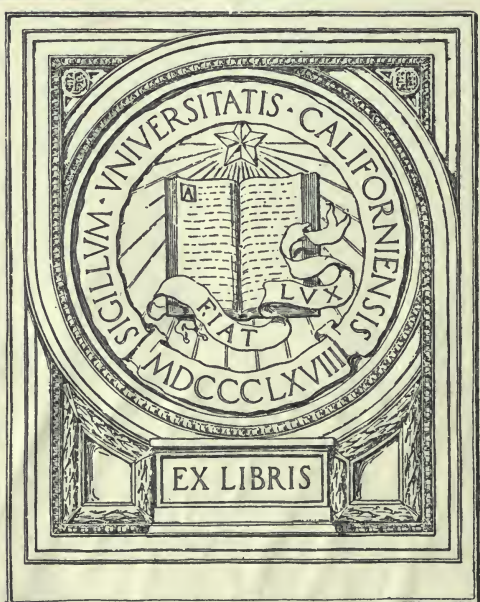
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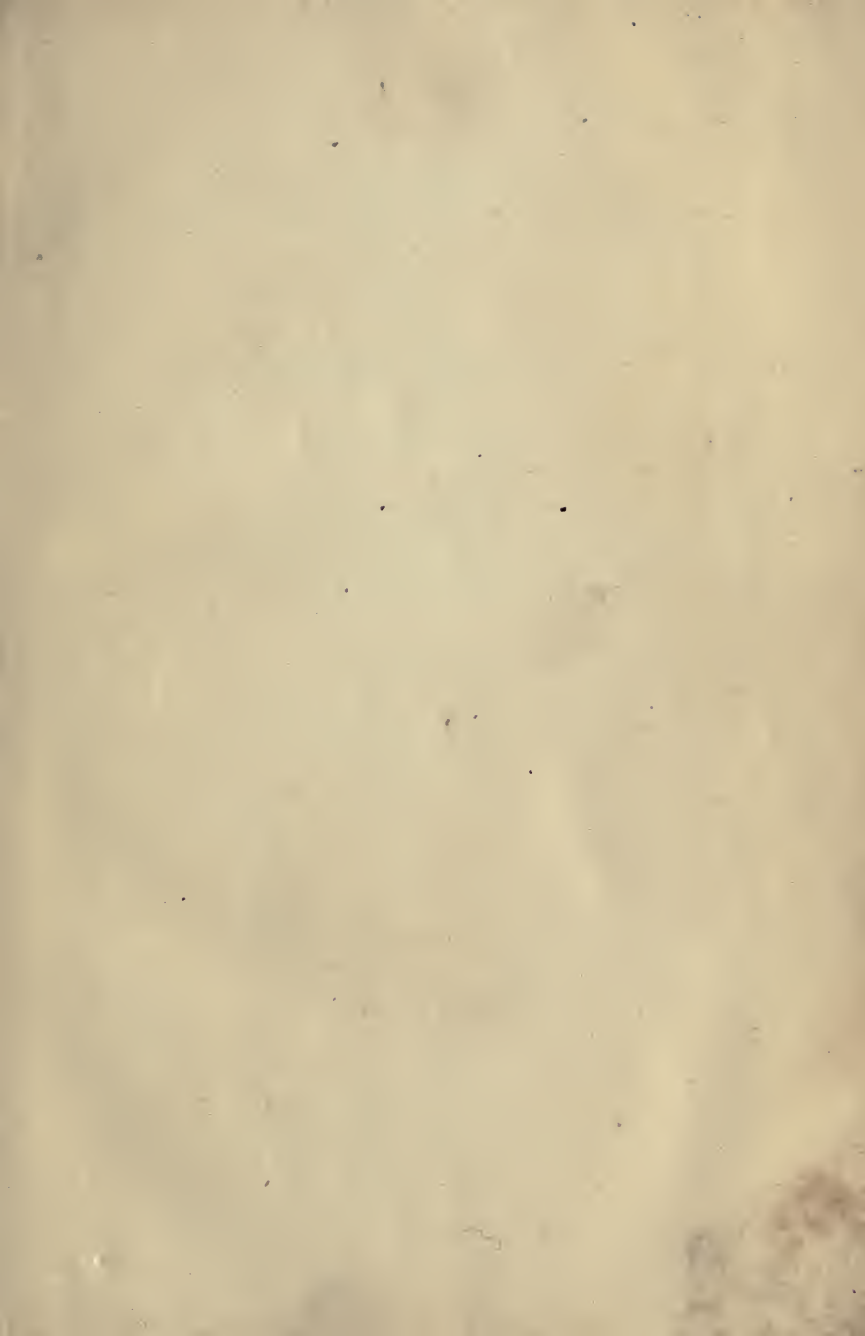
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ASSAYING.

IN

THREE PARTS.

PART 1ST.—GOLD AND SILVER ORES; PART 2D.—GOLD AND SILVER
BULLION; PART 3D.—LEAD, COPPER, TIN, MERCURY,
ZINC, NICKEL AND COBALT, ALSO, APPEN-
DIX TO PART 1ST AND 3D.

*Charles
new and*
BY C. H. AARON, METALLURGIST,

AUTHOR OF

"TESTING AND WORKING SILVER ORES," "LEACHING GOLD AND SILVER ORES."

PARTS 2 AND 3

PUBLISHED AND SOLD BY

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TO

The Mining and Scientific Press,

OF

SAN FRANCISCO, CAL.,

THE STEADFAST FRIEND OF TRUTH AND PROGRESS
—AN HONEST PAPER,

THIS BOOK IS RESPECTFULLY DEDICATED
BY THE AUTHOR.



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PREFACE TO PARTS II. AND III.

IT is not my desire to produce a book which shall supersede the necessity for thought on the part of the student. My intention is not so much to *teach* people as to aid them in learning. Some may prefer those books in which they find everything cut and dried, as it were; take so much of this and so much of that, do thus and so, without alternative and without exception, as though it were possible to give fixed and positive rules for all operations, in all cases. To such I would say, if they have not heads of their own, they would better turn their attention to something else than an art which must be practiced under ever varying conditions, and is based on a science in which new discoveries are being made every hour.

In the following, as in preceding pages, I have endeavored to make as simple and easy as possible the entrance to what, when properly appreciated and lovingly followed, must prove a pleasant and mentally profitable study, a pathway in which many beautiful flowers grow, but in which are also some thorns; in which none

can call himself an unerring guide, and in which but little real progress can be made without *hard work*. Assaying is a beautiful art, the parent, to a certain extent, and still more the offspring, of the sublime science of chemistry. The "practical" assayer, by which I mean him who has no other guide than his own limited experience, and such information as the writer of a hand-book can impart, may do tolerably well for a quartz-mill, or small assay office among the mines of gold and silver; scarcely for a smelting plant, and he will never realize the beauties, nor enter the upper story of the edifice in which he labors, until he has acquired a sound understanding of the fundamental principles of chemistry, as well as a very considerable acquaintance with its facts.

INTRODUCTION.

ALL the substances in nature are either simple or compound. A simple substance or body is one from which nothing different from itself can be extracted. There are sixty-four simple substances now known, and several more supposed. They are called elements.

The elements have a tendency to combine one with another, forming compounds; this tendency is called affinity. The strength of the affinities of different elements differs. The strength of the affinity of any one element for any other differs under different conditions of temperature, pressure, electricity, etc.

Most of the elements are known, and all are believed, to be capable of assuming the solid or the gaseous state; most of them also the intermediate condition of a liquid. The assuming of these different conditions by the same substance is dependent on temperature and pressure.

The elements may be *mixed* in *any* proportions, but they *combine* only in *fixed* proportions. When elements combine they form a substance which has different properties from those of the components. Two or more solids may combine to form a liquid or a gas: two or more liquids or gases may form a solid, etc. A knowledge of the proportions in which bodies combine, and of the conditions which determine their combination and dissociation, forms the basis of chemistry.

All the metals are elements. Their mixture by fusion forms alloys. Hydrogen, oxygen, nitrogen, chlorine, bromine, iodine, carbon, sulphur, selenium, phosphorous, boron, silicon, are elements which, with the metals, enter into the composition of the ores, fluxes, etc., which come under the notice of the assayer.

The fact that the elements combine only in fixed proportions, is the reason why a certain quantity of combustible matter produces only a certain quantity of lead from litharge in an assay. The element lead (a metal) combined with the element oxygen (a gas) in the exact proportion of 103.56 parts by weight of lead to 8 parts by weight of oxygen, forms 111.56 parts of litharge. Lead combines with oxygen, under certain conditions, in two other proportions, but the resulting compounds are not litharge, and the proportions are definite and unchangeable in each, combining spontaneously under proper conditions, and any excess of either element remaining separate.

The element carbon also combines with oxygen, 6 parts of the former and 16 of the latter, forming 22 parts of the gas commonly called carbonic acid gas (now called carbon dioxide). Under the action of heat, the affinity of carbon for oxygen is greater than the affinity of lead for oxygen. Hence if litharge and carbon are heated together, the carbon takes oxygen from the litharge and sets lead free. But this action can only take place between definite proportions of the substances. If twice 111.56 grains of litharge, containing 16 grains of oxygen, be heated together with 6 grains of carbon, the carbon takes all the oxygen, forming 22 grains of carbonic acid

gas, and the whole of the lead is set free; but if more litharge had been present that additional portion would have remained unchanged. From this we see that 6 grains of carbon reduce 223.12 grains of litharge, setting free 207.12 grains of lead, or 1 grain of carbon produces 34.52 grains of lead from litharge.

Hydrogen, sulphur, iron, and many other substances take oxygen from litharge under heat, in different but fixed proportions for each. Charcoal is slightly impure carbon, and 1 grain liberates only about 30 grains of lead from a corresponding quantity of litharge. Flour contains both carbon and hydrogen, but as it also contains oxygen and nitrogen it is less efficient, weight for weight, than charcoal, as a reducing agent: one grain liberates about 15 grains of lead. It is preferable to charcoal for assaying because it is cleanly and in fine powder.

Nitre consists of nitrogen, oxygen, and potassium. When heated it gives off a part of its oxygen, and if lead is present, litharge is formed, the quantity of lead so transformed depending on the quantity of the nitre in accordance with the law of definite proportions. If sulphur or carbon be also present, the oxygen combines with that, because the affinity is stronger than for lead.

Compounds can combine with other compounds, forming new compounds. The alkaline carbonates used in assaying are compounds of the metal elements sodium or potassium with oxygen and carbonic acid. Quartz (silica) is a compound of silicon with oxygen. When quartz is heated in contact with an alkaline carbonate, the latter is decomposed, not into its elements, but into carbonic acid gas, which escapes with effervescence, and sodium or potassium oxide, which combines with the quartz

—a compound with a compound—forming glass. Thus quartz, which is not fusible at any common furnace heat, is converted into an easily fusible substance.

One-fifth part of the air consists of oxygen, the other four-fifths of nitrogen. They are only mixed, not combined. Melted lead has a strong affinity for oxygen, which it takes readily from the air in such proportion as to form litharge. Gold and silver at the heat of melted lead do not combine with oxygen. Hence, when an alloy of lead, gold, and silver is melted in air, the lead oxidizes, the gold and silver do not. If the litharge is removed as it is formed, all the lead is finally separated from the precious metals. This is the philosophy of cupellation.

When silver is placed in nitric acid, it develops an affinity for a compound of nitrogen and oxygen which exists in the acid. Gold does not, hence when an alloy of gold and silver is boiled in nitric acid, the silver forms a combination known as nitrate of silver, which is soluble in water, and if the acid is somewhat dilute, all the silver is dissolved while the gold remains. From the law of combination in definite proportions, it is necessary that the acid be present in sufficient quantity, otherwise a portion of the silver remains unaltered. If more than the requisite quantity be used, the excess of acid remains unchanged. In practice, the alloy must contain at least twice as much silver as gold, otherwise the insoluble gold so envelopes the silver as to protect it from the action of the acid, except superficially.

Thus the *art* of assaying is dependent on the laws of chemistry, and although it may be practised with considerable success, it cannot be properly understood unless those laws are studied.

PART II.

GOLD AND SILVER BULLION.

In the assay of bullion, as in that of ore, the first step is to obtain a correct sample. For this reason it is desirable that the bars or ingots should be made in the establishment in which they are assayed. It is not often that an assayer will place his stamp on a bar without knowing to a certainty that the bar is what it purports to be. Moreover, the best sample is one that is taken from the molten metal, though this is not practised in the case of gold bullion, and it often happens that a lot of silver lead in bars, which in this country is called "base bullion," must be sampled without melting for the purpose. Silver bullion is sampled when melted, before casting. Silver lead is so sampled when practicable. Gold bullion, or base bullion in bars, is sampled by "chipping."*

Gold bullion is assayed by inquartation, involving cupellation, and parting (Part 1, page 74); silver bullion by cupellation (Part 1, page 66), or by the *humid method*; silver lead by cupellation, sometimes preceded by scorification.

Bars of bullion are called gold, doré, silver, or base.

*Silver bars are also chipped when the metal has been ladled into the moulds from a refining hearth.

The first consist almost entirely of gold, with a little silver and sometimes a very little base metal. They are stamped with the gold fineness and value only, the silver being allowed for in the market price. The second contain a large proportion of gold, a considerable proportion of silver, and sometimes a little base metal. Silver bars are such as consist mainly of silver with little or no gold, nor an excess of base metal. Base bars contain a large proportion of base metal, usually lead or copper. Dorée, silver, and base bars are stamped with the fineness and value of the gold and of the silver. Gold bars containing more than an insignificant percentage of base metal, although not enough to degrade them to the rank of base bars, are marked B, or Base, in addition to the gold fineness and value. Such are the rules of commercial assaying on the Pacific Coast, at least.

In order to extend the business of ore assaying to bullion assaying also, certain additional apparatus must be provided as follows:—

APPARATUS.

MELTING FURNACE.—A melting furnace suitable for light

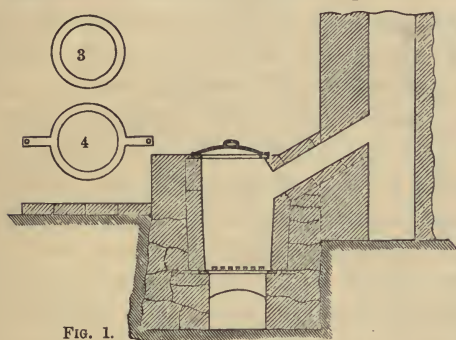


FIG. 1.

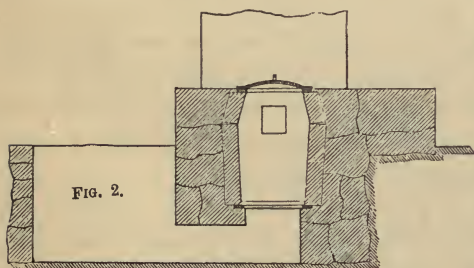


FIG. 2.

SCALE, $\frac{1}{4}$ INCH = 1 FOOT.

work may be bought ready made, and is similar to the crucible furnace for ore assays. For heavy work, such as casting large bars, it is best to build a furnace of masonry, and, though not customary, it is advantageous to have it sunk in the ground to within about a foot of the top.

The accompanying diagrams and description, from "Leaching Gold and Silver Ores," represent an excellent melting furnace, which the writer has built in silver mills.

This furnace is suitable for a number 30 or 35 black-lead pot, with charcoal as fuel. There is no heavy and expensive cast-iron plate on the top, and the cover may be of sheet-iron, although cast-iron is better.

The interior may be cylindrical, but is better, as shown in the figures, narrowed toward the top and bottom. The grate bars rest on an iron ring, Figure 3, supported by an offset in the masonry. The latter is mostly of a very rough kind, as the furnace is sunk in the ground to within a foot of the top, which, besides affording great convenience in working, obviates the necessity of iron bands or stay-rods to support it against the expansion caused by the heat. The greater part of the lining, 4 inches thick, is of good clay, very slightly moistened, and beaten round a hollow wooden core, for which part of a barrel of suitable size answers very well. It is topped with a course of common brick, and above these is a flat iron ring, Figure 4, with lugs, which may be bolted down, or simply let in flush with the top of the furnace.

The grate bars are not built in, but are free to be removed whenever desired. The furnace is constructed, when convenient, at the base of the main smoke-stack, with which it is connected by a flue; otherwise a large stove-pipe will answer for a chimney.

When the weight of melted metal does not exceed 100 pounds avoirdupois, the pot is lifted out of the furnace by hand, by means of the basket tongs, the mould being placed on the platform of masonry which is seen in the figure on one side of, and level with the furnace. The melter stands on the top of the furnace to lift, then steps to the ground one foot lower, to pour.

If larger quantities of metal are melted at once, a lever may be employed for lifting the pot. The lever is arranged like that of a blacksmith's bellows, being supported by a rope, chain, or swivel, in such a manner as to admit of a lateral as well as a vertical movement, and at a height of not less than six feet above the furnace, the center of which is directly under the shorter arm.

From the longer arm of the lever a rope depends; from the shorter arm a link of half-inch iron, long enough to reach, when drawn downward, into the cavity of the furnace, and terminating in a hook engaging in an eye-bolt, which forms the pivot connecting the jaws of the tongs.

The tongs being adjusted on the pot, a ring is slipped over the handles to hold them together; the melter steadies them, while an assistant, pulling on the rope at the other end of the lever, lifts the pot out of the furnace, and swings it near to the mould, when the melter pours the metal.

The hands and arms of the melter are protected by gloves, which, in some works, are elaborately made of canvas and padding. To make a glove, I simply take an ore sack, double it lengthwise, and sew it so as to form a narrow bag, of two thicknesses of canvas, into which the arm may be thrust to the shoulder. The gloves may be wetted to prevent burning, but hot articles must not be grasped with a wet glove, because the steam produced will scald the hand; yet moisture is a good protection against radiant heat, while grasping the cool handles of the tongs.

In melting with charcoal, the best result is obtained,

not by keeping the furnace full, but by letting nearly all the fuel burn away before refilling. A little practice will enable the melter so to manage that there shall be but little coal in the furnace when the time for pouring arrives, so that it is not in the way when seizing the pot with the basket tongs. The lifting must be performed without delay, otherwise the tongs may become red hot, and bend. The feet and legs of the melter may be protected by woolen armor, or by wetting the boots and trowsers; but melters and assayers must not shrink from a little scorching.

If there is much fuel left in the furnace when the melting is ended, the grate is taken out, and the embers fall into the ash pit, where they are extinguished.

Clinkers, if formed on the sides of the furnace, from the melting of the lining, or from dirty fuel, are punched off, while red hot, by means of an iron bar with a chisel end. If they were to be removed after cooling, the walls would be broken.

If making a bar of clean skimmed metal, it is proper to throw a little resin, or powdered charcoal into the pot, a few seconds before pouring. It prevents the sputtering which is often caused by the formation of base oxides. As soon as such a bar is cast, the top of it is covered with charcoal powder, to prevent oxidation while solidifying. When the slag or matte is poured with the metal the charcoal is not required.

In a first-class office, the top of the furnace consists of a heavy plate of cast iron, sloping upward toward the flue, and provided with sliding covers. A melting furnace should have a strong draft, controlled by a damper in the

chimney. The flue connecting the furnace with the stack or chimney should be 10 inches or more in width and about 6 inches high, in order to admit of placing a 6-inch cupel within it for a purpose which will appear hereafter. The floor, to a width of 4 feet on the front and sides of the furnace or set of furnaces, should be paved with bricks. A furnace of which the interior is 16 inches square, is large enough for the melting of 2,000 ounces of silver at once.



BLACK-LEAD CRUCIBLES.—Made of a mixture of clay and plumbago; used for the melting of bullion in the melting furnace; can be used from 10 to 20 times.

No.....	1	2	3	4	5
Prices, each.....	15c	20c	25c	30c	37c
Price, covers, ₣ doz.....		\$1.50	1.50	1.75	2.25
No.....	6	8	10	12	
Prices, each.....	40c	50c	55c	60c	
Price, covers, ₣ doz.....		\$2.50	2.50	3.00	3.60
Numbers 14, 16, 18, 20, 25, 30, 35, 40, 45, 50, 60, 70, 80, 90, 100, 150.					

Price, per number, 4½ cts. Covers, price, per number, 2½ cts.

TONGS.—Straight, bent, and basket tongs of several sizes, adapted to the crucibles. The basket tongs are constructed to grasp the pot around below the bulge,

or both above and below, by curved arms attached to the jaws; they are used only for the larger sizes of pots, say above number 20. Smaller pots are seized by the rim with the straight or crooked tongs. Also bar tongs with broad, flat, and long jaws for lifting hot bars. Straight pokers for adjusting the coals, and bent for clearing the grates from below, are needed.

SKIMMERS.—Iron rods 3 to 4 feet or more in length, of $\frac{1}{4}$ to $\frac{1}{2}$ inch thickness; at one end formed to a close flat spiral, making a disc from 2 to 4 inches diameter at an obtuse angle to the rod, which is suitably bent for convenient use in removing slag from the surface of molten metal.

GLOVES.—Made of canvas, with a thumb, but not separate fingers; padded on the back. Used to protect the hands of the melter from the heat. A narrow bag of doubled sackcloth, or blanket covered with canvas, into which the arm can be thrust to the shoulder, is as good, and much cheaper.

STIRRERS.—Strips of the same material as the crucibles. Pieces sawed from the sides of wornout pots answer. Used for stirring the molten metal.



BAR MOULDS.—Made of cast-iron. Used to pour the molten metal into in order to form a bar or ingot.

Ounces, Silver,	15	20	27	40	52	80	107	112
Ounces, Gold,	28	35	50	75	107	150	200	207
Price, each,	\$.50	.50	.50	.75	1.00	1.25	1.75	2.00

Ounces, Silver,	160	210	250	265	320	428	500	638
Ounces, Gold,	300	388	400	495	600	800	921	1000
Price, each,	\$2.75	3.25	3.50	3.75	4.00	4.25	4.50	5.00

Larger sizes can be had up to 2,000 ounces of silver.

HAMMERS AND CHISELS.—Round faced hammers for smoothing bars, and chipping hammers for driving the chisel, of different sizes, from 4 ounces to 4 pounds, according to the bars. Thin sharp cold-chisels for chipping.

SUNDRIES.—A strong table should stand near the melting furnace, on which to place the crude bullion, as gold-dust, or lumps of retorted amalgam, etc., in sheet-iron pans, in readiness for melting; also to place the cleaned bars on, etc. There should be a rack in which tongs and fire-irons may rest, standing on end on the bricked floor, conveniently for use. A simple rail with some nails projecting will serve. Some put the nails in the edge of the table which then stands behind the melter, who has only to turn around and put his hand on what he requires; also a tub of water in which to cool the bars, with a sloping platform adjoining, on which to clean them, arranged to drain into the tub; a box of sharp sand for scouring the bars, a scrubbing brush, and a tight roll of canvas about $1\frac{1}{2}$ inches thick, the wetted end to be dipped in the sand and applied to the bars as a scourer. For polishing gold bars some use a bundle of brass wires as a brush. Diluted sulphuric acid, in a tub or vat, is sometimes used for "pickling" a bar containing base metal, to improve its appearance, and a bottle of acid is usually kept at hand for use in the scouring. A block of timber set on end, the top covered by sheet zinc and enclosed by a rim or ledge, is required to place bars on for chipping. Some small sheet-iron

pans for samples (*chips or dips*), and larger ones for crude metal.

STAMPS.—Letters, figures, dollar sign, No., Oz., and words, such as Fine, Value, Total, Gold, Silver, and the name of the assayer; made of steel and used for marking the bars. Sizes, from $\frac{1}{8\frac{1}{2}}$ inch to $\frac{3}{4}$ inch. Should be arranged in holes in a block of wood for convenience.



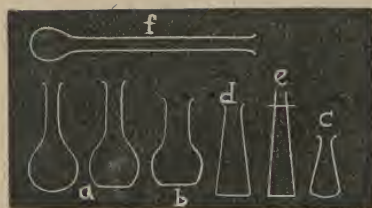
BULLION BALANCES AND WEIGHTS.

Becker's Balance, 100 oz. to $\frac{1}{2}$ grain and weights.....	\$ 55.00
“ “ 500 “ “ 1 “ “ “	195.00
“ “ 2,000 “ “ 2 “ “ “	338.00
Oertling's London, 200, 300, 1,000, 2,000 ozs.	

Glass cases and supporting tables are supplied, if required.

BULLION SCALES.—These scales, though strong and massive, are also delicate, and are provided with apparatus by which the respective bearings are relieved when not in use; also with an arrangement by which the pans are steadied while the weights are adjusted. In using the balance, the object to be weighed and the heavier of the necessary weights must be placed on the pans before the beam is suspended on its bearings by a turn of the crank in front of the base. The lighter weights for completing the weighing are then adjusted while the pans are steadied by the arrangement spoken of above, and released by a touch when desired. Scoops and pans for gold-dust, etc., accompany the scales.

In large establishments, two pairs of bullion scales are used, one very massive pair for large quantities of metal (chiefly silver), and a lighter pair for smaller lots, especially gold. The larger balance has only the upper part inclosed in a glazed case; the gold scales are completely inclosed. In silver mills, platform scales are often used for the weighing of the bars; they can be obtained to show both troy and avoirdupois weight, or the latter may be converted into troy by calculation, or by means of the Assay Table for 20 grammes of ore in Part I.

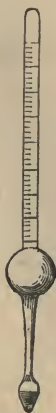


MATRASSES.—Thin glass flasks, used for parting bullion assays, and for other purposes. Three ounce, flat-bottomed matrasses are suitable for bullion parting.

No. 20.	Round Bottom, per doz.	\$1.50	1.75	2.00	2.25	2.50
	Capacity in oz.	1	1½	2	2½	3
No. 21.	Flat Bottom, per doz.	\$1.25	1.25	1.50	1.75	2.00
	Capacity in oz.	¼	½	1	1½	2

CHARRED LENTILS.—Made by heating lentils in a closely covered vessel until no fumes are given off. Boiling acid is liable to *bumping*; this is prevented by placing a small piece of charcoal in the matrass. Common charcoal answers, but charred lentils throw off no fragments.

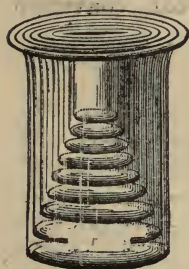
HYDROMETER.—A floating gauge, made of glass and loaded with shot at the lower end. Placed in a liquid it floats in a vertical position, a greater or less portion of the graduated stem being submerged according to the density of the liquid; used as a measure of the density, and therefore the strength, of acids, etc. There are several kinds of hydrometers; that of Baumé is generally used in assay offices. In ordering a hydrometer, state the purpose for which it is required, because some are arranged for liquids lighter than water. These would not answer for acids. It is best to procure one which is graduated to show specific gravity as well as degrees Baumé, because the strength of the acid is indicated in either in the books. Price, 75 cents.





GRADUATED CYLINDER.—The hydrometer is used in a narrow glass vessel, which need not be graduated for this purpose, but a graduated vessel is useful, and it answers equally for floating the hydrometer. As to the graduation, grammes and c. c. are the same, a gramme being the weight of a c. c. of pure water at standard temperature.

Contents in cubic cent.....	5	10	25	50	75	100
Open top with spout, price, each .	\$0.30	.40	.50	.65	.75	1.00
With Glass Stoppers				\$0.75	.90	1.00 1.25
Contents in cubic cent.....			250	500		1,000
Open top with spout, price, each ...			\$1.25	2.00		3.00
With Glass Stoppers, price, each.....			\$1.62	2.25		3.50
With spout reading upwards) graduated into grammes, {	5	10	20	50	100	200
Price, each	\$0.30	.40	.50	.75	1.00	1.25



BEAKERS.—Thin glass jars, which will bear rather rapid change of temperature without fracture. Used in treating substances with acids, etc., and for receiving the liquid from a filter (*filtrate*). Not essential in the ordinary routine of assaying gold and silver, but very appropriate in an office, and requisite in the assays of

copper and other base metals.

Nest of	3	5	6	8	10
Price each nest, plain.....	\$0.38	.75	1.25	1.60	3.00
“ “ with lip		\$1.00	1.50	2.00	3.12

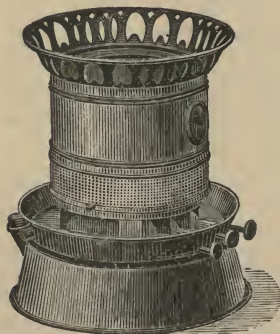
SINGLE BEAKERS—PLAIN.

In depth, inches	10	8	6	6	5½	4½	4	3
In width, inches	6	5	5	4	4	3½	2½	2
Price, each	\$1.00	.90	.80	.60	.50	.40	.25	.15



EVAPORATING DISHES.—Made of glazed porcelain. Will withstand heat and acids; used for dissolving and drying; not essential in assaying gold and silver.

No.	00	0	1	2	3	4	5	6	7
Capacity, oz.	2	4	8	16	24	1 qt.	3 pt.	½ gal.	¾ g.
Diameter, in.	3½	4½	5½	6½	7½	8½	10	11	12
Price	\$0.30	.35	.40	.60	1.00	1.25	1.35	1.50	1.80



COAL-OIL STOVE. — Useful anywhere. Almost indispensable in a country office where gas cannot be had. A sheet-iron dish containing some sand, placed on the stove, forms a sand-bath convenient for the parting.

No. 00.	One Burner.	Price, each	\$1.75
No. 1.	" "	"	3.20
No. 2.	Two Burners	"	4.20
No. 3	" "	"	5.30
No. 4.	Three "	"	6.50

FLATTING MILL (Rolling Mill).—A pair of steel rollers, with adjusting screws and cranks to operate by hand. Used in laminating metal; can be dispensed with, but not conveniently, by using the hammer and anvil instead. Prices, from \$35.00 to \$65.00. Other apparatus will be mentioned in the course of description of methods.

MELTING BULLION.

THE fuel used is coke or charcoal, or in some places, stone coal. It should be screened free from dust and dirt. Lumps larger than a turkey's egg should be broken. When coke is used, a little charcoal is necessary to start with.

New black-lead pots must be annealed. To anneal a pot and cover, kindle a fire in the furnace, and invert the pot over the fire; put the cover also in the furnace; add fuel, check the draft, and increase the heat very gradually until the pot and cover are red-hot. They are then ready for use, and may thereafter be placed, even when cold, in the hottest fire without danger of fracture.

The metal to be melted, if in small quantity, is put into the cold (annealed) pot as it stands on the table, or on the furnace plate; the pot is then placed in the fire, resting on a piece of fire-brick. Otherwise the pot is put into the fire first, and large bars or lumps are placed within it by means of tongs, gold-dust or small fragments of metal by means of a scoop and sheet-iron funnel. Borax is added, the pot covered, and fuel placed around and above it. Retorted amalgam, especially silver, is porous and bulky; hence the pot must be refilled from time to time as the metal subsides, and as such metal contains some quicksilver, the addition should be

made before that in the pot is quite melted, while it is in a pasty condition, otherwise spirting may occur. The melter should be careful not to inhale the fumes.

When the metal and the slag formed by the borax with the impurities are melted, the latter must be removed. The skimmer is placed in contact with the slag, of which a portion adheres; the skimmer is then withdrawn, pressed on the furnace plate previously greased, or on any suitable surface, to chill and flatten the adhering slag, dipped slightly in cold water to further cool it, and again touched to the slag. If the slag is in such quantity as to accumulate inconveniently on the skimmer, it is thoroughly cooled in water and knocked off by a blow with the hammer. Toward the end, however, it is better if the skimmer is not cleaned, because the metal is less liable to adhere to a surface of slag than to the naked iron.

The slag must be of medium consistency, neither pasty nor too liquid. If pasty, it may retain considerable globules of metal; if too thin, it cannot be readily skimmed off. A thick slag may be thinned by an addition of borax; or if sand be the cause, a little soda may be employed; but as soda attacks the pot, it must be used with care. A thin slag may be thickened by means of sand.

When all slag has been removed, if the surface of the metal is bright it is ready for pouring. If it is covered by a scum or crust, it probably contains copper or iron, perhaps also sulphur. If rainbow colored rings appear, lead is most likely present in small quantity, and more if brilliant spots appear moving rapidly.

In the first case, some lumps of borax are to be thrown on the metal and allowed to melt, absorbing the base oxides and forming a slag which must be removed as before. If this does not seem to be efficacious, sulphur is probably present, and a little nitre may be added to the borax, or better a mixture of nitre and glass powder. If much copper or iron is present, it is best to add a little lead to the metal in the pot; the colored rings or bright spots then appear, being produced by the formation of litharge, which rapidly oxidizes the copper and iron, forming slag. As the plumbago of the pot is a reducer (Part I, page 33), it constantly tends to the reconversion of the litharge to lead, and of other oxides to the respective metals, while litharge, being a flux for all earthy matters, attacks also the clay of the pot. To prevent this reverse action and protect the pot, some cupels are introduced which float on the liquid metal and absorb the litharge, etc. The cupels are removed when saturated. Unless the bullion is very nearly all precious metal, the refining by these methods is too tedious, but sulphur should be removed in order to make a bar which can be correctly assayed.

While the melting is in progress, a stirrer is heated to redness, and a suitably sized mould is warmed. To prevent adherence of the metal, a mould for a gold bar is smoked by being placed in a small chamber, or mounted on two brickbats, over a dish of burning resin, or a quite small mould, by holding it over the flame of a lamp. It is thus warmed at the same time. For a silver bar of any considerable size, greasing is usually preferred; or the mould may be painted with a wash of finely sifted ashes and water; then thoroughly dried and warmed.

After the skimming, the metal in the pot is covered by a layer of charcoal powder, and left for a few minutes, the pot being covered. This reduces any base metal oxide that may have formed, and which would cause the metal to sputter when poured, or it prevents the sprouting of nearly pure silver. A silver, doreé, or base silver bar, if of considerable size, must now be sampled (all very small bars are chipped). The pot is taken from the fire, placed on the furnace top, and the metal stirred by an assistant, with the heated stirrer held in the tongs, while the melter dips out two separate samples of 30 to 50 grains each, and pours them, from a height of a foot or two, into dishes of water, by which the metal is granulated. The dipping is usually done with a very small crucible, hessian or french, heated to redness and held in a pair of tongs. A heated rod of iron is often used for the stirring, and, if the end is formed into a small ladle, for the dipping also; an assistant is not then required. The metal is then poured into the bar-mould, the charcoal being held back, if necessary, by means of an iron, such as the bent poker, until nearly all the metal has run out. Some more charcoal dust is then thrown on the bar to keep the surface covered; or, if the bar is small, some oil may be poured on it. The purpose is to prevent oxidation. The slight cooling of the metal during the stirring and sampling does no harm, for, if silver is poured too hot, the face of the bar will be spongy. The granulated samples, or "dips," are dried, and are then ready for the assayer. A gold bar is poured as soon as melted and stirred, the samples not being taken by dipping.

In the melting rooms of Professor Price, in San Fran-

cisco, the heaviest bars are cast without the use of a crane or of basket tongs. Two men grasp the pot on opposite sides by means of straight "side tongs," the jaws of which are formed to suit the thickness of the pot, so as not to bite a piece out, and it is lifted out of the furnace in less time than would be required to adjust the basket tongs. The pot is tilted for the pouring by means of a pair of tongs, which are held in a horizontal position, having semi-circular jaws grasping the pot about the middle. This way of pouring requires considerable strength, and the basket tongs, suspended from a crane by means of an eye formed on the pivot of the tongs, and having the handles secured by a ring slipped over them, seem preferable for heavy work. At Los Bronces, in Mexico, the furnace was deeply set in the ground, the ash-pit covered by an iron grating on which the workmen stood. The pot contained enough metal for a number of bars. It was lifted by a crane, and placed in a carrier, such as is used by founders for carrying melted iron to the flasks, and two men lifted it and filled the moulds in succession.

When the bar is solid, it is turned out of the mould by upsetting that, cooled in water, any slag beaten off, scoured, smoothed if necessary by hammering, and dried. A bar which has been sampled by dipping is now ready for weighing, otherwise it must first be chipped. The chips are taken from two diagonally opposite corners, one top and one bottom. The chisel is held between the thumb and forefinger of the left hand, while the remaining fingers of that hand retain the chips which would otherwise be projected to a distance. The chips are taken in many small pieces, to facilitate

the weighing of the assay, and each sample is placed on a separate piece of paper, one corner of which is bent up for convenient handling. The number of the deposit is marked on the paper. One assay is made from each sample, and if the two do not agree, and a "control" assay fails to correct the discrepancy, the bar is remelted and better mixed. The samples should be large enough to allow of several assays being made of each, and any residue is folded in the paper, the number being left visible, and kept for future reference in case of need. The chipped corners of the bar are neatly dressed by the hammer, and impressed with a stamp, as a security against being tampered with. The bar is then weighed, and a tag bearing the number and weight pasted on, to remain until the stamping; or the number and weight may be stamped at once.

Each bar is finally stamped on the face, which is that side which was the bottom when cast, with the name and address of the assayer, the weight in troy ounces and hundredths, and the fineness and value in gold, or in both gold and silver, according to the character of the bullion, as explained above. On delivery it is accompanied by a certificate, stating the number of the deposit, the name of the depositor, the weight before and after melting, and all other particulars as stamped;

C. H. AARON,	
ASSAYER.	
No.....	1754
Oz.....	172.54
Fine Gold.	.978
Value,	\$3,488.24

also the charges for melting and assaying, against which is offset the value of the *chips* and *grains*. The chips are the samples; the grains are any

globules of metal which may remain in the pot after

pouring, or which may be removed with the slag in the skimming, all of which must be collected, cleaned, and weighed. A convenient way in which to arrive at the weight of the chips and grains is to weigh the bar and grains together before chipping, then chip, and weigh the bar alone for its delivery weight. The difference is the weight of chips and grains, the value of which is reckoned according to the result of the assay. The value stamped on a bar is the assay value. The commercial value depends on the state of the market for the particular description of bullion.

Gold is frequently purified from base metal in the following manner: The melted metal is slowly poured from a height of several feet into a vessel containing water, which is meanwhile agitated by means of a stick. The metal is thus granulated. It is then dried and remelted with addition of nitre and borax. The melting with nitre must not be done in a black-lead pot, but in a "sand-pot," or one of clay. The nitre in decomposing leaves caustic potassa which violently attacks the pot, as do also the base metal oxides formed. This may easily lead to fracture, when the spilled metal must be recovered, as far as possible, by washing the ashes and cinders, after allowing the fire to burn out. To prevent such an accident, two devices are employed. Firstly, a dish, made by cutting down a wornout black-lead pot, is placed under the sand-pot in the furnace, as a saucer under a cup. This receives the metal in case of a break. Secondly, a quantity of clean quartz sand, equal to about half the bulk of the nitre, is mixed with that and the granulated metal. The sand combines with the

metal oxides and the potassa, forming a liquid slag or glass. Without this addition of sand, iron or copper oxide would not become liquid unless a large proportion of borax were used. The metal and slag are poured together into a mould. Sometimes a very neat bar is thus made, which does not require remelting, but frequently the slag gets between the metal and the mould, spoiling the appearance of the bar, and making it difficult to clean. In this case the bar is remelted, with a little borax in a black-lead pot. If, however, it is not pure enough, the granulation and melting with nitre are repeated. Retorted amalgam does not require granulation, being porous enough for the melting nitre to permeate it.

The fusion with nitre must be done carefully and under a very low heat for some time; otherwise the contents of the pot will overflow. The pot should not be more than half filled with the mixture. It is not safe to attempt a second use of the sand-pot, as it is almost certain to break.

A very good way in which to purify small lots of gold or silver containing base metal is by cupellation. A worn out black-lead pot is cut down so as to form a dish from 2 to 4 inches deep in which a cupel is made by beating in moistened bone ashes, and scooping a hollow in the top. The cupel is well dried in the dish, which may have some holes drilled through it with advantage. When required for use, it is placed in the flue of the melting furnace while the fire is in use for other purposes. As soon as the cupel is red hot, some lead is placed on it, and when that is melted the impure gold or silver is added and

cupelled without expenditure of time or much fuel, because the melting of other lots may be going on in the furnace. The furnace must be left a little open to admit air for the cupellation.

A few ounces of metal may be cupelled in the ordinary muffle, a cupel being made in the following manner: a strip of cotton cloth is laid across a small bar-mould, and pressed down into it, the ends of the cloth projecting. Moist bone ashes are then beaten in until the mould is full. A depression is formed in the top by means of a knife, and smoothed by the bowl of a spoon. The cupel is then lifted out of the mould by means of the strip of cloth which passes under it, and dried.

Refining as described does not remove silver from gold bullion. In Australia, silver and copper are removed from gold by injecting chlorine through a clay tube (pipe stem) plunged into the melted metal. The chlorine combines with the copper and silver; the copper chloride volatilizes; the silver chloride floats on the gold and is poured off as soon as the gold solidifies by cooling, after which the gold is again heated to melting and cast in a mould.

ASSAYING BULLION.

IN ore assaying (Part I) the weight of precious metal obtained is usually stated in thousandth parts of a gramme, of a half gramme, or of ten grains, regarded as integers and so written, to which I have given the name of "points," a term borrowed from the bullion assay, and more suitable than "thousandth," which expresses a fraction. In bullion assaying, unfortunately, nomenclature and notation are not quite consistent. The object of the assay is, to find out how many parts in one thousand equal parts by weight of the bullion consist of pure gold or silver; or, which amounts to the same, mathematically, how many .001 of 1 part are gold or silver. In writing, and in stamping the bar, the latter view is taken, and we use the decimal mark thus, fine gold .572; fine silver .422; that is, so many thousandths of a part in one part of the alloy. But in speaking we say, the bullion is so many fine in gold or silver, not mentioning thousandths, but implying so many parts (units) in a thousand. The mathematical value of the two modes of expression is identical. Moreover in speaking the thousandths are often called "points," as, "this bar is four points finer than that," or, "add three points for cupellation loss."

To show the difference between the reporting of an ore assay and that of a bar; supposing the gramme weights to

be used, 0.065 gramme, or 65 milligrammes of gold, if got from an ore assay would be written (and called) 65, but if from an assay of bullion would be written .065; the bar would be stamped, fine gold .065; but in speaking of it we should say, "It is sixty-five fine," adding, "in gold," if that was not already understood. In either case it would be meant that one thousand parts, of whatever denomination, consisted of sixty-five parts gold and nine hundred and thirty-five parts some other metal.

From this it will be seen that either of the systems of weights used in ore assaying will answer for bullion also, or indeed any set of weights decimally divided down to one-thousandth of the greater weight, and it matters not whether the greater weight be regarded as the unit, and the lesser as a fraction of that, or the lesser be considered the unit, and the greater as one thousand of such.

For gold bars, using a delicate balance, half a gramme is generally taken for the assay, the purified metal being weighed in half milligrammes. With an inferior balance, or for a silver bar, one gramme or ten grains are the usual quantities. Whatever it be, the weight taken is called a thousand, whereas, to be consistent with the written expression, it should be called one, and the thousandth part of it, which to agree with the term thousand ought to be called one, is called a thousandth, or a point, which is better. Hereafter in these pages, we shall speak of thousands and points, discarding the awkward word thousandth entirely.

In bullion assaying, all the weighing, except that of the lead used, is done on the assay balance.

GOLD BAR.—Natural gold is never pure. It is al-

loyed almost invariably with silver, frequently with copper, and the bars often contain platinum and iridium which occur in grains with the placer gold, lead, from bullets scattered about the gravel washings, and quartz-mill gold sometimes contains iron from the abrasion of stamps. All base metal can be removed by cupellation with lead; silver and platinum, by nitric acid; for, although pure platinum is not attacked by nitric acid, it dissolves in that when alloyed with two parts of silver; and as gold must also be alloyed with at least twice its weight of silver, in order that the parting may be complete, it will be readily seen that the presence of platinum offers no serious difficulty. Iridium does not melt to an alloy with the gold, but remains in the form of scales or grains.

It will be remembered that, while two parts of silver to one of gold are indispensable to a successful parting, more than three parts would cause inconvenience by the crumbling of the gold. Moreover, it has been ascertained that, with more than three parts, the separation is less perfect than with two and a half parts, until the proportion of eight parts of silver to one of gold is reached, when the gold falls to absolute powder and remains pure.

The assayer endeavors to obtain an alloy for the parting, in which the proportion of silver to that of gold shall be about two and a half to one, supposing platinum to be absent. A practiced assayer can usually judge from the appearance of the sample, or from former experience with gold from the same locality, how great a proportion of silver it already contains, and, consequently, how

much must be added; otherwise a preliminary assay may be made with addition of so much silver as to assure the success of the parting, even though the gold may crumble, and thus the requisite information be obtained.

PRELIMINARY ASSAY OF GOLD BAR.—One thousand of the sample and four thousand of test silver are enveloped in three or four thousand of sheet-lead, and cupelled under a rather high heat. The bead is cleaned, beaten by hammer and anvil to a flat plate, annealed by heating to redness, parted, cupped, dried, and weighed, as in ore assaying, but a matrass is used for the parting, instead of a test-tube, on account of the larger quantity of metal. The weight in points is the approximate fineness from which the weight of silver required in the proper assay is known by multiplying by 2.5.* From this the quantity already alloyed with the gold must be deducted, in order to ascertain how much must be added. For example, if the gold got from 1,000 of the sample weighs 924, the lost weight is that of the silver, except, perhaps, a very small quantity of base metal, which need not be noticed in this class of bullion. The bullion then contains 924 of gold, and 76 of silver in 1,000. Multiplying 924 by 2.5, we have 2,310 of silver required for the parting, of which quantity the bullion already contains 76, leaving 2,234 to be added.

ASSAY PROPER OF GOLD BAR.—One thousand of the sample is cupelled with such a quantity of test silver as is necessary, about 3½ points of copper and about 3,000 of sheet-

*In the case of very small bars, this assay will suffice, if the gold be twice boiled in acid, well washed, cupped with care, and at least two points deducted from the result found.

lead, rolled into a capsule or "cornet" (Part I, page 76), and closely folded about the assay. The lead cornets are usually kept at hand, ready made. The weighed assay is transferred directly into the cornet, held in the left hand, from the weighing capsule which is lifted from the scale pan with the pincettes. It is not usual to weigh the silver for the inquartation. A quantity of sheet silver is cut into pieces of nearly known weight, and the required number of pieces, as well as the copper, added to the gold in the lead cornet, which is folded in the form of a snug packet, and placed on a watch-glass, or in a dry cup, which occupies its appropriate place on a tray according to the set number of the assay.

As many as 14 assays may be thus prepared and cupelled at one time in a mint muffle. The cupels are heated to light redness, and the assays are placed on them in regular order, according to their respective numbers in the set. The muffle is then closed until the assays *uncover*, when the door is left open. If there is any difficulty in keeping the front cupels hot enough, some glowing coals may be placed in the mouth of the muffle. It sometimes happens, in cupelling a number of assays, that the uncovering of one or more is delayed by some cause; a small wad of folded paper, placed on the refractory assay will set it working. A strong heat is maintained throughout the operation, and especially at the time of brightening, that is to say, a higher degree than is admissible in a silver assay, because gold is less volatile than silver.

It is not necessary to remove a gold assay the instant the cupellation is finished, so that all may remain in the

muffle until all are finished, and may then be taken out without much precaution as to cooling, as these assays are not very liable to sprouting; the addition of copper is partly for the purpose of preventing the sprouting, for, notwithstanding the cupellation, a trace of it remains in the bead. Another reason for the addition of copper is that it renders the metal tougher for the lamination. If, for want of heat, an assay should *freeze*, or solidify, during the cupellation, it may be refused by placing glowing coals about the cupel and one upon the top of it; but the assay is not quite reliable.

The cupelled bead is cleaned, beaten to the form of a bar about one-quarter inch wide and one-eighth thick, annealed by heating to dull redness in the muffle, and passed through the flatting mill until it is reduced to a strip about as thin as an ordinary card. The metal must be annealed whenever it shows signs of cracking on the edges, or becomes hard and elastic from the rolling, and again after the rolling is finished. It is then formed into an open coil* by the thumb and finger, placed in a three-ounce matrass and *gently* boiled in one and a half ounce of nitric acid of 32° Baumé, adding a charred lentil, or a bit of charcoal to prevent bumping. After thus boiling for 10 minutes, the acid is poured off, replaced by a fresh quantity, and the boiling repeated for 15 or 20 minutes. It is in the second boiling that the charcoal is especially necessary, as there is more danger of the bumping of the acid when the evolution of gas ceases.

The acid is now again poured off, and this time the charcoal must go with it, or, on the addition of water, it

*The name of "cornet" is given to this as well as to the lead capsule.

will sink and give trouble; the gold is washed three times with hot water, cupped, heated to redness in the muffle, cooled, and weighed. It should be in one piece, which, dark colored and fragile after the parting, assumes the golden hue and a fair degree of tenacity when heated. The color, although golden, is liable to considerable variations of shade.

The weight of the gold in points, less from half a point to one or two points allowed for a small portion of silver which remains in it and is called the *surcharge*,* is the gold fineness of the bullion, which is stamped on the bar. The results of two assays should agree within a quarter point in weight.

DORÉE BAR.—The assay is conducted, speaking generally, in a similar manner to that of a gold bar. A preliminary is more often necessary in this case, and according to its indications either gold or silver may be added so as to produce a suitable alloy for the parting. If gold is required, it is taken from the cornets of previous assays, or pure gold may be bought for the purpose.

In the assay proper, if gold is required it is weighed, added at once to the weighed assay, and the whole cupped with a suitable quantity of sheet-lead. The difference between the weight of the bead, and that of the assay *plus the added gold*, represents base metal. The bead being laminated, parted, etc., the further loss of weight is the silver fineness to which a correction may be added, according to the judgment and experience of

* A larger surcharge is allowed for very small bars than for large ones; also when a little base metal is believed to be present, the bar being brittle, etc.

the workman, for cupellation loss, from one to three or more points. Finally, the weight of the cornet, less that of the added gold and a suitable allowance for surcharge, is the gold fineness. Except in particular cases, the in-quartation by addition of gold is generally neglected.

If an addition of silver is necessary, as for instance in the case of a bar which is about half gold, it, like the gold, may be added at once, and allowed for in the final estimation of the silver fineness. It is perhaps better to cupel the assay with the requisite quantity of lead to clear it of base metal; note the weight of the bead, then add the silver and cupel with a very light lead cornet, laminate, part, etc., and weigh the gold. The difference between the gold fineness and the weight of the bead after the first cupellation, plus a suitable correction for loss, is the silver fineness. By operating in this way the cupellation loss of the added silver need not be considered.

Example: A thousand of dorée bullion containing some copper, was cupelled with 12,000 of lead, and gave a bead weighing 900. It being supposed to contain too much gold for parting, 2,000 of silver were added, and the whole wrapped in lead and again cupelled, then parted. The gold weighed 400. Deducting 400 from 900 left 500 for the approximate weight of the silver. In the assay proper $400 \times 2.5 = 1,000$ of silver was requisite; the bullion contained 500, hence 500 of silver must be added.

One thousand of the bullion was then cupelled with 8,000 of lead, and gave a bead weighing 907. This was again cupelled with 500 of silver and a small lead cornet,

then laminated, parted, etc. The gold was in a single piece and weighed 401. The bar was therefore 401 fine gold, and $907 - 401 = 506$ fine silver, to which a point might be added for cupellation loss. Unless, in particular cases, or for quite large bars, the process indicated for the preliminary, if executed with skill and judgment, answers all purposes.

BASE BAR.—This assay is better scorified before cupellation. An approximation to the quantity of lead required, and to the gold and silver fineness, may first be obtained by cupelling 1,000 of the bullion with as much lead as may be found necessary for clearing the bead. Beginning with the quantity of lead which is judged to be required, and watching the operation, more lead can be added from time to time until the bead is seen to round up, and to exhibit the brilliant play of colors immediately preceding the brightening. Silver is to be added, if supposed to be requisite for the parting; it must be weighed. In the assay proper, 1,000 of the bullion may be scorified with the proportion of lead found necessary in the preliminary, and a little borax. When the scorification has been carried far enough, the assay may be poured, or cooled in the scorifier and that broken, the button placed on a hot cupel, and any desired addition of gold or silver made. The gold or silver must be weighed, wrapped in sheet-lead, and added to the button after that is melted; or, the addition of silver may be deferred until the bead is cleared, cleaned, and weighed, as in the assay of a dore bar, and for the same reason, yet if the bullion contains much copper, and but little silver in proportion to the gold, it is better not to

postpone the addition of silver, because, in the cupellation of gold and copper, without a considerable proportion of silver, some gold is lost. For this reason on the other hand, the addition of gold when much copper is present is better postponed until the copper has been removed. Neither gold nor silver should be added during the scorification as needless losses are incurred thereby, all of which will inure to the detriment of the bullion. It has been observed by experimenters, that if gold is added to lead already melted on a cupel, little or no loss occurs, but if the gold be previously scorified with lead, and then cupelled, there is a notable loss. As with dore bullion, the inquartation by addition of gold is not generally practiced. Where so much lead is used as sometimes in the assay of base bullion, it need not be all sheet-lead; pieces of assay bar-lead may be used on the cupel, and either that or granulated lead in the scorifier, so that sheet is only required for wrapping the assay and the gold or silver for the inquartation, for which it is safer than the paper recommended by some writers, though paper may be used in case of need.

Example of assay of base bar containing lead and copper:—

Preliminary cupellation without addition,	
gave a coppery bead, weighing	732
Showing that the assay contained about	268 lead
Recupelled with 10 parts =	7320 lead
Did not clear; added 2 parts =	1464 lead
Which cleared the bead. Total lead	8784

The bead weighed.....	327
Added an equal weight of silver; cupelled and parted, gold weighed.....	47

Silver.....	280
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In the assay proper, 1000 scorified with 8500 of lead and cupelled with addition of $\frac{280}{2.5} - 47 =$

65 gold, weighed..... 396

Parted; gold weighed..... $112\frac{1}{2}$

Silver weighed..... $283\frac{1}{2}$

True weight of gold (after deduction of 65 added from the total)..... $47\frac{1}{2}$

The assay therefore shows the bar to contain in 1

part, fine gold..... 0.0475

“ silver..... 0.2835

Base metal..... 0.6690

1.0000

If the proportion of gold to silver had been reversed, the bead from the preliminary would have been yellow. We should then have added twice its weight of silver in the preliminary (see Part I, page 76), and 2.5 times 280 of silver in the assay proper.

The following table, from Mitchell, will give an idea of the proportion of lead required to separate copper from gold by cupellation:—

GOLD IN ALLOY.	LEAD REQUIRED.
.900.....	10 parts
.800.....	16 "
.700.....	22 "
.600.....	24 "
.500)	
to {	
.050)	34 "

For alloys of copper and silver, about half the above proportions of lead suffice, but in both cases much depends on the temperature of the muffle and on other conditions. The guide is the condition of the button; if it is hard, brittle, or coppery, it must be treated with a fresh quantity of lead.

GOLD AND PLATINUM.—The presence of platinum in the assay is known by the following signs: The brightening of the bead requires a much higher heat than usual, and is less vivid. The bead is flat, thick edged, and dull. On parting, if the platinum is in the proportion of 1 to 50 of gold, the acid is colored straw yellow. The annealed cornet is paler than usual, even to resembling tarnished silver. It is important to recognize the presence of platinum in gold bullion, lest it be mistaken for silver, because, while not removed by cupellation, it is dissolved in the parting. In order that the parting may be effected in the presence of platinum, the quantity of silver alloyed must be as much as twice the weight of the gold and platinum together, and as both the silver and the platinum are dissolved in the ordinary parting with nitric acid, it may happen that the gold will crumble.

An alloy of gold and platinum with 12 parts of silver may be parted by boiling in sulphuric acid, leaving the gold and platinum, the silver alone being dissolved. The gold and platinum are washed with hot water, dried and weighed; then again alloyed with $2\frac{1}{2}$ parts of silver and parted with nitric acid, leaving the gold, which is washed, dried, and weighed. The more usual method is to make an alloy with enough silver to part in nitric acid, which leaves the gold; then to precipitate the silver from the liquid, in the form of chloride, by adding hydrochloric acid. The silver chloride is separated from the liquid by filtration or by decantation (pouring off), dried and weighed; 75 per cent. of the weight is silver, from which the weight of the added silver must be deducted, leaving the silver fineness of the bullion. To the liquid containing the platinum is added a little hydrochloric acid, and then solution of ammonium chloride as long as a precipitate forms. The liquid is then evaporated to dryness, and to the residue, when cold, dilute alcohol is added, and the insoluble portion (platinum ammonium chloride) collected on a filter, washed with alcohol, dried and heated to redness. The residue is platinum, which is weighed. The united weight of the gold and platinum, deducted from that of the cupelled bead (minus any added silver), leaves the silver fineness of the bullion, which should agree nearly with that found by calculation from the precipitated chloride as above.

Iridium does not form an alloy with the gold. It mostly falls out of the cornet during the lamination, in the form of scales of a steely color. If in large quantity, a part may be found at the bottom of the pot in which the bullion is melted.

SILVER BAR.—Silver bullion usually contains copper or lead, or both. The proportion of lead required to remove the copper depends on that of the copper to the silver. If the quality of the bullion is not approximately known, it may be ascertained by a preliminary cupellation with as much lead as may be found necessary. The assay proper is then treated accordingly. Any lead which the bullion may contain is counted as a part of that required, hence if the bullion contains some lead, 1,000 of it may be cupelled without other addition than the small piece of sheet-lead necessary to envelop it. If the bead clears, no further addition is required; if not, lead is added in successive small portions until it does, and the quantity of lead so added is approximately that which will be requisite in the assay proper. A table will be given which will aid in the apportionment.

If much lead is required, it is best to melt the greater portion of it on the cupel before adding the assay; bar-lead should be employed in this case. When the lead is quite melted, the assay is added, enveloped as usual in a lead capsule, the weight of which makes up the required quantity. In order to make a proper correction for the cupellation loss, a *test assay* is made at the same time and in the same heat. Test silver and copper are weighed off in the proportions in which they exist in the bullion, as nearly as may be judged, or as indicated by the preliminary if made, and cupelled with the same weight of lead as is used for the assay. The loss sustained by the test, or for safety, a point less, is added to the result of the assay. Unless the bar is quite small, two assays, one from each of the two samples, should be made, and should agree within a point.

The cupellation must be conducted with great care as to heat. I have known as much as 30 points to be lost by unskillful cupellation, and, although a test or check assay may be made with each bullion assay, or in the midst of a group of those, of approximately similar quality, yet, when losses are excessive the check cannot be implicitly relied on for the correction. Tables of the loss for bullion of various grades of fineness are given in books, but are only useful as showing what has been, and therefore can be, done. The loss should seldom be so much as 10 points, and with bullion that is nearly fine should not exceed 3.

The smoke from the cupels should rise to about the middle of the muffle, the draught through that not being so strong as to draw it immediately through the holes, a part generally escaping by the front. If much lead is used, crystals of litharge should form on the cupel at a little distance from the bead, and even the brightening heat should not cause them to disappear. This is called "feathering" the cupel. When the bullion is so fine as to require but little lead, it is scarcely possible to feather the cupel without great risk of freezing the assay, and an assay once frozen, even though remelted, is not reliable.

On account of the delicacy of this operation and the occasional necessity for moving a cupel during its progress, as well as at the finishing, only a small number of assays can be properly made at once in a muffle which is only 4 or 5 inches wide. When the bead is about to brighten, it should be pushed further back where the heat is higher, or a glowing coal may be placed before it; it

must remain in this higher heat only a few seconds after the brightening, say while 5 can be deliberately counted; it must then be cooled with the precautions indicated in Part I, page 67, the best portion of which, being a method not generally published, may be repeated here.

The cupel is drawn to the front of the muffle, and gently tapped on one side by the tongs. At the instant when the bead ceases responding to the taps by its vibration, and a certain change, which will be recognized when once seen, comes over it and the face of the cupel, indications of the solidification of the bead, it is pushed back into the hottest part of the muffle, and there left for a minute or thereabout, after which it will not sprout, being solidified all through, as shown by a dimple on its surface caused by contraction. The heat must not be so intense as to remelt the bead, or the effect desired will be lost, as will also some silver; but a bead is not very easily remelted when once chilled. The bead must be white, well rounded, *slightly* adherent to the cupel, and crystalline at the bottom when cleaned. It is weighed, and then parted without addition of gold to inquartate. Any gold found is cupped, dried, weighed, giving the gold fineness, which, deducted from the weight of the bead, leaves the silver fineness to which is added a correction indicated by the check. The following table from Mitchell gives the approximate proportion of lead required for the cupellation of different alloys of silver with copper:—

SILVER IN ALLOY.	LEAD REQUIRED.
1.000 (pure silver)	.300
.950	3.000
.900	7.000
.800	10.000
.700	12.000
.600	14.000
.500 to pure copper	17.000

The reason that 1,000 of pure silver requires 300 of lead is that silver alone cannot be melted on the cupel without its throwing off minute globules, nor can a good bead be formed without the aid of lead; so that no matter how nearly pure the silver may be, that quantity of lead, at least, must be used.

SILVER LEAD.—This assay is very simple. It is only necessary to wrap the weighed assay in a lead capsule, in order to ensure the union of all fragments, and cupel it with the usual precautions. A better way is to take several thousands of the sample, and scorify with a little powdered glass or quartz, repeating, if needful, until the button is of a suitable weight, and then cupel. The bead should be parted, and results must be divided according to the number of thousands used for the assay.

The value of silver lead, in precious metal, is usually reported as so many ounces of gold and so many of silver in the ton, hence an assay ton (Part I, page 80), or any quantity commonly used for ore assays may be taken, being weighed on the pulp scales, scorified and cupelled, and value calculated as for ore. Or, the fineness in points divided by 10 is the percentage from which ounces or values per ton can be reckoned. (See Part I, *Ib.*)

TO FIND THE VALUE OF A BAR.—The weight of the bar in troy ounces multiplied by the gold fineness (stated in thousandths), gives the weight of pure gold contained, the value of which is computed at \$20.67 per ounce. The weight of the bar, multiplied by the silver fineness (stated in thousandths), gives the weight of pure silver contained. Silver is reckoned at \$1.2929 + per ounce.

The above values of pure gold and silver, multiplied by the fineness of the bar in each metal (stated in thousandths), gives the value per ounce of such bullion for each metal; and the weight of the bar, multiplied by the respective values per ounce, gives the value of the entire bar for each. It is well to make the calculation in both ways, in order that the one may check the other.

The dealers in assayers' goods distribute, gratuitously, sets of tables showing the value per ounce of bullion of any fineness in gold or silver. The value per ounce being found from these tables, it only remains to multiply the weight of the bar in troy ounces by that.

The value per ounce of bullion of any fineness may be found by means of the "Assay Table for One Assay Ton of Ore" on page 106 of Part I, with more accuracy than by calculation, unless a great number of figures be used. One ounce of bullion 1,000 fine is of the same value as 1,000 ounces 1 fine, or 372 ounces 1,000 fine equals 1,000 ounces 372 fine, and so of any fineness. Hence, to find the value of 1 ounce of bullion of any given fineness, consider the points of fineness as so many ounces 1,000 fine, and find the value of that quantity as directed for an ore assay. Divide by 1,000 (move the decimal point); the result is the value of one ounce.

Example: What is the value per ounce of silver bullion .840 fine ?

$$\begin{array}{rcl} 800 \text{ ounces } 1,000 \text{ fine} & = & \$1034.34 \\ 40 \quad \text{"} \quad \text{"} \quad \text{"} & = & 51.72 \end{array}$$

\$1086.06

This divided by 1000 gives \$1.086, the value of 1 ounce. A similar process gives the value for gold.

To find the value of any number of ounces of bullion of any fineness by the same table, multiply the weight by the fineness; the result is the weight of pure metal, gold or silver as may be. Find the value of the pure metal as directed for ore assays.

Example: A silver bar weighs 1,642 ounces and is .840 fine. What is its value ?

$1,642 \times .840 = 1,379.28$ ounces of pure silver; then from the table:—

1,000 ounces.....	\$1292.93
300 " 	387.88
70 " 	90.50
9 " 	11.63
0.2 " 	0.26
0.08 " 	1
1379.28	\$1783.21

This use of the table is much facilitated when it is arranged so that two figures can be operated on at once, in which form I have all these tables; but they, especially the gold tables, contain too many figures for this book, being calculated to 9 and 10 places from a valuation of \$20.671834625323 per ounce of gold.

It often happens, in the country, that a bar must be weighed in avoirdupois pounds. One pound avoirdupois is equal to 7,000 grains troy, or, $14.583 +$ ounces. Multiplying the pounds and decimal fractions by the latter number converts them into troy ounces. The number of troy ounces corresponding to any number of avoirdupois pounds may be found very simply and accurately from the assay table for 20 grammes of ore, on page 104 of Part I, by proceeding as there directed; or, the value of a bar, of which the weight is given in pounds, may be got without finding the ounces. Again, the value of pounds and ounces avoirdupois may be computed by the aid of the following table, after multiplying the weight of the bar by the fineness, in gold and silver respectively, for the contained weight of the pure metals.

	GOLD.	SILVER.
1 pound.....	\$301.46.....	\$18.85
1 ounce.....	18.84.....	1.17
$\frac{1}{4}$ "	4.76.....	0.29
$\frac{1}{8}$ "	2.38.....	$0.14\frac{1}{2}$
$\frac{1}{16}$ "	1.19.....	$0.07\frac{1}{4}$

HUMID ASSAY OF SILVER BULLION.

OWING to the uncertainty attending the fire assay of silver bullion, the humid method is used in all United States mints and first-class private offices. This method depends on the fact that silver in nitric acid solution is completely precipitated in the form of chloride by a solution of common salt. At the same time the salt is decomposed, so that, if suitable proportions of silver and salt be thus brought together, neither can afterward be found in the liquid.

In the Royal Mint of India, the precipitated silver chloride is dried and weighed, and from its weight that of the pure silver is calculated. In this country and in Europe, the quantity of silver in the solution is estimated by the quantity of salt solution of known strength requisite for its precipitation. The salt solution is either weighed or measured. The latter method is the most general, and is called the *volumetric assay*.

A solution of salt in water is made, of which 100 cubic centimeters will exactly precipitate 1,000 of pure silver, leaving neither salt nor silver in the liquid. This is the *normal solution*. The thousand used in this assay is either 10 grains or 1 gramme, the latter generally. Another solution of salt is made one-tenth as strong as the normal; consequently 1 cubic centimeter of it precipitates

one point of silver. This is the *decime salt solution*. A decime solution of silver is also prepared, of which 1 cubic centimeter contains one point of silver; consequently the two decime solutions are equivalent, volume for volume, each neutralizing the other. The sign for a cubic centimeter is c. c.



MEASURING THE NORMAL SOLUTION.—A glass *pipette* containing 100 c. c. is used. The accompanying diagram and directions are from Mitchell. “Immerse the jet (*c*) of the pipette in the solution, apply the mouth to the upper orifice, and draw the liquid into *d*, above the circular mark, *ab*. Dexterously apply the forefinger of one of the hands to this orifice, remove the pipette from the liquid, and hold it as represented in the figure. The mark *ab* is held on a level with the eye, and the surface of the solution allowed to descend until it forms a tangent with the plain *ab*. At this instant the jet *c* of the pipette is set at liberty by removing the finger against which it had been pressed, and, without otherwise changing the position of the hands, the contents are allowed to run into the bottle appropriated for that purpose” (containing the assay),

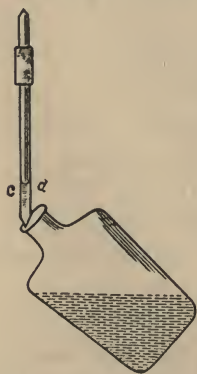
“taking care to remove the pipette as soon as the stream stops.”

“If, after having filled the pipette by aspiration, there is any difficulty found in a sufficiently rapid application of the forefinger to the superior orifice to prevent the fall

of the liquid below the mark *ab*, the pipette must be removed from the liquid, the orifice being closed by pressing the tongue against it; then apply the middle finger of one of the hands to the lower orifice, remove the tongue, and apply the forefinger of the other hand to the larger orifice, previously wiped dry." The pipette should be rinsed with a little of the solution before being charged, and washed twice with water after use.

MEASURING THE DECIME SOLUTIONS.—The decime solutions are measured in smaller pipettes gauged so that, when filled to the mark *cd*, they allow 1 c. c. to run freely, the small quantity of the solution remaining in the pipette not forming part of the c. c. The decime salt solution is kept in a bottle, closed by a cork which is traversed by the pipette firmly fixed in a hole bored for that purpose. The decime silver solution is kept in a bottle closed by a glass stopper. Quoting Mitchell again:—

"To measure a thousandth ('point') with the pipette, the bottle is held with one hand, and the pipette with the other. The pipette is taken from the solution after its upper orifice has been closed by the forefinger; the lower orifice is then inclined against the edge of the flask to remove the liquid, which, without this precaution, would remain there; the mark *cd* is then raised to the level of the eye, and, by a suitable pressure of the forefinger on the upper orifice, which may be obtained by giving the pipette a slight alternating circular movement between the fingers, the solution is allowed to run out gradually.



The instant the concave surface of the liquid is at the level *c d*, the pipette is firmly closed by pressure of the forefinger on its orifice, which is held above the bottle into which the solution is to be poured, and the forefinger removed so that it can be emptied. It is here necessary to remark that in order to regulate the slow and regular runnings of the liquid from the pipette, by the pressure of the forefinger, the latter ought to be neither too moist nor too dry; if too dry, it will not perfectly close the orifice, even by strong pressure; if too moist it prevents the entrance of air, and the liquid will not run or if it does it will be irregularly." (This observation should not be lost sight of in the use of the large pipette.) As it is often convenient to use several c. c. of either decime solution at once, the pipettes are graduated from 1 to 5.

PREPARATION OF THE NORMAL SOLUTION.—One thousand of silver requires $541.66+$ of salt, hence 1 gramme of silver requires 0.5417 gramme, or 10 grains require 5.417 grains. It is more troublesome to prepare pure salt and to measure pure water with perfect accuracy, than to make a solution which approaches the required standard, and then rectify it by an addition of salt or water, as indicated by the result of an assay of pure silver, or silver of known fineness, made with it.

The easiest way in which to prepare the approximate solution is to make a *saturated* solution of good table salt, and to dilute a certain quantity of that to the required strength. Take about a pound of salt and dissolve it in about four times its weight of water. Filter the solution, and boil it down to a quarter of the original

quantity, or less, causing the deposition of the greater part of the salt. Pour off the liquid, and wash the salt quickly with water. Redissolve the salt in as little water as will suffice, and again evaporate until a little of it is again deposited. Cool the liquid and put it into a bottle. The liquid is a saturated solution containing 26.5 per cent. of its weight of salt, as nearly as can be ascertained. One hundred c. c. of this solution, at 60° F. contains enough salt for 49 assays if the thousand be 1 gramme, or for 75.5 assays if the thousand be 10 grains. Therefore, for the gramme system take 1 litre (or 10 charges of the large pipette) of this solution, and add 48 litres of water to make normal solution for 490 assays; or, take 2.08 volumes of the saturated solution, and 97.92 volumes of water. For the grain system take 1 litre of saturated solution to 74.5 litres of water to make normal solution for 755 assays; or take $1\frac{1}{2}$ volume of saturated solution and 98.66 volumes of water. If more convenient, take for the gramme system 5 parts by any kind of weight, of saturated solution, and 189 parts by weight of water, or 1 to 37.88. For the grain system, 1 part, by any kind of weight, of the saturated solution to 58.89 of water.

If preferred, pure salt may be made by neutralizing pure hydrochloric acid with pure sodium carbonate or bicarbonate, evaporating to dryness and heating almost to redness. The mass should be powdered and kept in a stoppered bottle. Nearly pure salt may be prepared by dissolving table salt in water, boiling until a large proportion of the salt is deposited, pouring off the liquid, washing the salt twice with a little water, and drying,

first between sheets of blotting paper, and then by heating almost to redness. In making normal solution from the dry salt, take for each litre of water 5.417 thousands of salt, which is 5.417 grammes or 54.17 grains according to the weights used for the assays. The most suitable way in which to operate is to place the salt, weighed for the required number of litres of solution, into the litre flask, and add a sufficient quantity of water to dissolve it easily; when the salt is completely dissolved, fill the flask to the mark with water, and empty it into a carboy; again fill the flask, and pour the water into the carboy, as many times as may be necessary to make the desired quantity of solution. Or take 10 pounds avoirdupois weight of water and

for 1 gramme = 1,000—24.61 grammes

“ 10 grains = 1,000—246.0 grains

Of salt. Dissolve the salt in a small portion of the water and add the remainder.

The solution, in whatever way prepared, must be thoroughly mixed. This may be done by rolling and shaking the containing vessel, or by stirring with a wand. The plan recommended when dry salt is used, of dissolving it first in a small portion of the water, makes it easier to be sure that it is *all* dissolved than if it were added to the whole quantity of the water in the larger vessel. The mixing of the solution with the rest of the water is not difficult.

PREPARATION OF THE DECIME SALT SOLUTION.—Any volume of the normal, diluted with 9 such volumes of water makes a decime solution. Measure with the large pipette 100 c. c. of water, which pour into the appropri-

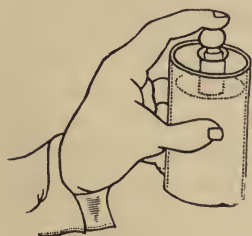
ate bottle; remove 10 c. c. of the water, by means of one of the small pipettes, and replace it by an equal volume of normal solution; mix by shaking; this small quantity is for use in correcting the standard of the normal; after this has been done, a new decime solution must be prepared if the error of the normal was considerable, and a larger quantity will be required. To prepare it measure 1 litre* of water, or weigh 1,000 grammes at 60° Fahrenheit; remove 100 c. c. of the water, and replace it by 100 c. c. of the corrected normal solution, by means of the large pipette.

PREPARATION OF THE DECIME SILVER SOLUTION.—One thousand of pure silver is dissolved in about 7 c. c. of nitric acid in a litre flask, and the flask is then filled to the mark with distilled water; or at least with water which does not cause the slightest turbidity. If a litre flask is not at hand, 1,000 grammes of water (15,432 grains) at 60° Fahrenheit may be weighed, a volume of it equal to that of the silver solution removed by means of a pipette, and the remainder added to the silver solution. This quantity will suffice for a very considerable number of assays, and as it will inevitably become stronger by evaporation if long kept, it may be as well to make a smaller quantity; it is only necessary that each c. c. of it shall contain a point of silver and not more.

STANDARDIZING THE NORMAL SOLUTION.—A thousand of pure silver is dissolved in about 10 grammes of nitric acid of about 32° Baumé, in a bottle of clear glass

*A litre is 1,000 c. c.

which will contain about 200 c. c. To facilitate the action, the bottle is placed in a vessel containing warm water. When the silver is dissolved, the red fume which fills the bottle is blown out by means of an inserted glass tube so bent as that the workman's face may not be over the bottle while blowing. As soon as the assay has cooled, 100 c. c. of normal solution are added by means of the pipette, and the bottle is put into a japanned tin case, or enveloped in a cloth, to prevent the action of the daylight on the silver chloride, which might slightly vitiate the result, *and the stopper, previously dipped in pure water, is put in. The bottle, held as shown in the



diagram, is briskly shaken for several minutes, which causes the precipitate to curdle, and settle rapidly, leaving the liquid perfectly clear. On placing the bottle on the table, to allow of the settling, a brisk to and fro circular movement is given to it, so as to wash down from the sides any adhering chloride. When the liquid is quite clear, the stopper is removed and suspended in a clamp, or bracket, so that the moistened part may touch nothing. A c. c. of decime salt solution is poured into the bottle. If, immediately or after a few seconds, a cloudiness is perceptible in the liquid, it is evident that the normal solution is weak, because 100 c. c. of it have not precipitated 1,000 of silver. The liquid must be again

*This precaution will be unnecessary if the room is illuminated by windows of yellow glass.

cleared by shaking, and another c. c. of decime salt solution added, and so on until no further cloud is produced. A record of the number of c. c. of decime solution used is kept, but the last one is not counted, because it produced no effect, and the one before the last was probably not all required, so it is counted as a half. Supposing that 7 c. c. caused precipitation, but the 8th did not; only $6\frac{1}{2}$ must be counted.

It now *appears* as though 1,006 $\frac{1}{2}$ points of silver had been present in the solution, but it is known that there were but 1,000, because that quantity was weighed; the normal solution is therefore $6\frac{1}{2}$ points weak, and requires a corresponding addition of salt. The quantity of salt required is found by the following:—

RULE.—Multiply the weight or measure of salt or saturated solution used in making the entire quantity of normal solution by the number of c. c. of decime salt solution which were *necessary* to complete precipitation, and divide by 1,000. The result is the weight or measure of the required addition.

As a portion of the normal has been withdrawn for the test and for making the decime solution, the quantity thus found may be corrected by the deduction of a corresponding percentage; but it is not worth while, unless either many tests have been made, or but a small quantity of normal has been prepared, for, even though the addition required may be considerable, it is evident, from the fact that the normal is weak, that the saturated solution used was less strong, or the salt less pure, than was supposed, so that the added quantity will have the same deficiency in proportion. If dry salt was used in

making the normal, and consequently will be used also in the correction, the required quantity should be dissolved in some of the original normal poured into a beaker for that purpose, and then mixed with the main stock, the beaker being rinsed with a very little water and that also added. Another test must then be made, and, if necessary, another correction.

If the first addition of 1 c. c. of decime salt solution produces no precipitate, it is known that the normal is either correct or too strong. To ascertain which, add 2 c. c. of decime silver solution; one of these is consumed in neutralizing the c. c. of decime salt previously added, hence only one is counted. Shake down, and add another c. c. of decime silver; if this produces no cloud, the normal is correct within at most a point, and may be accepted, for it is needless to make it perfect, for reasons which will appear hereafter. If a cloud is produced, shake down again, and continue adding decime silver solution and shaking down until the last added c. c. causes no cloudiness; then, as with the salt, reject the last c. c. and half of the preceding one. Supposing that, in all, 5 c. c. have been used; we reject one of the first two added, because it only counteracted the c. c. of decime salt previously added; we also reject the last, because it gave no precipitate, and the one before the last we count as a half c. c. Thus we have $2\frac{1}{2}$ c. c. of decime silver solution which were necessary to complete the decomposition of the salt in 100 c. c. of the normal. Apparently only $997\frac{1}{2}$ points of silver were dissolved, but we know that 1,000 were so; consequently the normal is $2\frac{1}{2}$ points too strong, and water must be added. The

quantity of water required is found by the following:—

RULE.—Multiply the quantity, by weight or measure, of water used in making the entire quantity of normal solution by the number of c. c. of decime silver solution which were *necessary* to complete the assay, and divide by 1,000. The result is the quantity of water to be added to the normal solution. Mix thoroughly.

A correction for the percentage of the normal which has been used is more necessary in this case; still it is hardly worth the trouble of making it because, if the error of the normal is large, a second rectification will probably be required, and if it is small, the difference will not be perceived. After mixing, test again; when the normal is sufficiently exact make a new decime salt solution.

EXAMPLES.—The normal solution was made with 5 lbs. avoirdupois of saturated solution, and a test assay on pure silver required $8\frac{1}{2}$ c. c. of decime salt solution to finish; then, 5 times $8\frac{1}{2}$ make 42.5, and this divided by 1,000 gives 0.0425 lbs. of saturated solution to be added to the normal. A pound avoirdupois is equal to 7,000 grains troy, and multiplying this by 0.0425 we have 297.5 grains.

Again, the normal solution was made with $189\frac{1}{2}$ lbs. of water, and proves to be too strong, a test assay requiring $5\frac{1}{2}$ c. c. of decime silver solution to finish; then, 189.5 multiplied by 5.5 gives 1,042.25, which, divided by 1,000 gives 1.04 pounds of water to be added.

If dry salt had been used, or if the saturated solution or the water had been measured by volume, the same kind of calculation would give the required result in terms of the weight or measure used.

When the normal solution is within about a point of being correct, it may be accepted, perfect accuracy being needless on account of the changes of volume produced by alterations of the temperature, for which a correction must be made as hereafter directed, and includes any inaccuracy in the standardizing; for the present, however, the solution will be supposed to be exact.

THE ASSAY.—For the sake of convenience, a c. c. of either of the decime solutions, will be called a *point of silver*, or, a *point of salt*, as the case may be; also 100 c. c. of the normal solution will be called a *thousand of salt*.

One thousand of salt requires 1,000 of silver, and such a weight of the bullion is taken for the assay as contains, as nearly as may be, 1,000 of silver. This being dissolved, and 1,000 of salt added, we then proceed, by means of the decime solutions, to ascertain how many points more or less than 1,000 the assay contains. Knowing the weight taken, and having found the weight of the contained silver, it is easy to find by proportion what weight of silver 1,000 of the bullion contains, which is its fineness.

In order to be able to take the required weight of the sample, we must have an approximate knowledge of its silver fineness. This is obtained by means of a preliminary assay by cupellation and, if necessary, parting. As we know from experience that there is a certain loss of silver in the cupellation, we add to the result of the preliminary assay such a number of points as the judgment may dictate. From the approximate fineness thus obtained we calculate the weight which must be taken in

order that the assay may contain nearly 1,000 of silver; thus, suppose the cupellation (and parting) gave a silver fineness of .864, and we add 3 points for loss, making .867, then, as 867 are to 1,000 so are 1,000 to 1,153.4, and we weigh off 1,154; hence the

RULE.—Divide 1,000,000 by the number of points of approximate fineness; the result is the desired weight in points.

Dissolve that quantity of the sample in about 7 c. c. of nitric acid, in the manner directed for the standardizing of the normal solution; cool, add 1,000 of salt, shake down and add a point of salt. If a cloud is produced, proceed to complete the assay with salt, as in testing the normal solution, keeping a record of the number of points added, finally rejecting the last and half of the preceding one. Suppose 1,154 of the bullion to have been taken, and that 3 points of salt have given a precipitate, the 4th none. We have then found that 1,154 points of the bullion contain $1,002\frac{1}{2}$ points of silver. To find the fineness, we take the proportion, as 1,154 are to 1,002.5 so are 1,000 to 869.7. As not less than a point is usually stamped on a bar, we may say the bullion is .869 fine.

If the first added point of salt gives no precipitate, add a point of silver to neutralize it, shake down and add another; if this produces no precipitate, the assumed fineness was correct; if otherwise, proceed to finish with silver in the same manner as with salt; then, rejecting the first point because it was required to counteract the point of salt, and the last because it gave no result, also half of the preceding one because it was probably only

required in part, deduct the remainder of the added points from 1,000, which leaves the number of points in the assay. If, as before, 1,154 of the bullion were taken, and one added point of salt gave no precipitate, and 4 successive points of silver gave a precipitate, the 5th none, then it is known that 1,154 of the bullion contain $998\frac{1}{2}$ of silver, and as 1,154 are to 998.5 so are 1,000 to 865.25, or the bullion is .865 fine. Hence, to find the fineness, the following

RULE.—Multiply the points of silver found by 1,000, and divide by the points of bullion taken.

Greater exactitude can be attained by finishing with half points, but for ordinary purposes it is not necessary. The gold fineness of the bullion is found in the preliminary assay by cupellation and parting.

It is better to finish with salt than with silver, so the weight of bullion taken for the assay should be such as to contain a little more, rather than less than 1,000 of silver; and if it is not so, and is found to contain less, a number of points of silver may be added at once, so as to make more than 1,000; the assay is then finished with salt. An account must be kept of the silver and salt added, the one +, the other —. If more than eight points of salt or silver are required to finish, it is advisable to make another assay on the basis of the result found.

The last trace of precipitate is somewhat difficult to discern. It can be seen best by looking obliquely upward through the liquid, while holding the bottle toward the light. Another help is to have a black background, for which a black felt hat answers. The writer and some

others have found it advantageous to have the assay solution tinged by copper; the white silver chloride shows very plainly in the blue solution. An experienced assayer, on observing that the first added point of salt causes a considerable precipitate immediately, will add several more points before shaking down, being able to judge about how many will be required; he can also tell, almost with certainty, when another point will be without effect.

In case the furnace is not fired, a preliminary assay may be made by means of the blowpipe. It is not necessary that any special weight be taken, therefore select a piece of the bullion which may weigh about 100, more or less; weigh it, cupel it with lead before the blowpipe flame, not allowing the flame to touch the metal, but heating the cupel around it by directing the point first on one side and then on another; weigh the bead, part, and weigh the gold if there is much; then, as the weight of the silver is to that of the assay, so is 1,000 to the weight of the bullion to be taken for the volumetric assay, to which a few points may be added for the loss, which is likely to be greater, at least in inexperienced hands, than in the muffle. The gold may be calculated for 1,000 on the same principle, but it may be better to dissolve 1,000 of the bullion in nitric acid, and weigh the gold from that, the only difficulty being that it is likely to be in finer powder than when parted from a bead which has been purified from base metal by cupellation.

An excellent preliminary, and even an accurate assay, may be made by means of a Mohr burette.* Dissolve

*For a description of this, and its use, see Part 3d.

1,000 of the bullion as usual. Charge the burette very accurately with normal solution, and run that into the assay bottle, first in larger, then in smaller quantities, finally drop by drop, shaking down each time, until the last drop or two produces no precipitate. The value of a drop may be ascertained by noting how many drops lower the float 1 c. c. The last addition, which produces no precipitate, is not counted, and the preceding is counted for half its value. The burette should be read after each addition toward the end. It should be of 100 c. c. capacity, graduated to tenths, and furnished with a float. If, by accident, an excess of salt solution is run in, add ten or more points of decime silver solution, making a note of it; and then finish with salt. If preferred, the assay may be finished with decime salt solution from a second burette, or from the small pipette, but with care this will not be necessary, as the normal can be measured to less than a point. The number of c. c.* necessary for complete precipitation is the fineness, provided no addition of decime silver has been made; otherwise that must be deducted. Special care must be taken that no air remains in the jet of the burette when charged, to which end it is best to partly charge it from below, by suction, and then fix it in the stand and fill it from above.

CORRECTING THE ASSAY.—It has thus far been supposed that the normal solution was accurately standardized so that 100 c. c. would exactly precipitate 1,000 of silver. In order that this may be the case, it must not only have been made of the correct strength, but it must always be used at the same temperature. This cannot

* This should have been written tenths of c. c.

well be. For this reason a *check assay* is made, on pure silver, with each set of bullion assays, a number of which can be conducted at the same time, and are corrected according to the error found, whether that is caused by expansion or contraction of the solutions, owing to a change of the temperature, or from inaccurate standardizing.

The result of an assay of pure silver should be 1,000 fine; if it gives more or less, the assay of each sample of bullion must be corrected in proportion to its fineness. This is arrived at with sufficient exactness, by adding to, or subtracting from, the number of points found in the assay, the amount of the error of the check, *before* calculating the fineness of the bullion. Thus: if the result of the check is $2\frac{1}{2}$ points *above* 1,000, that quantity must be deducted; if below, added. Suppose the check was $2\frac{1}{2}$ points high, and 1,154 of bullion gave $1,005\frac{1}{2}$; we deduct $2\frac{1}{2}$ from that, leaving 1,003, from which we then calculate the fineness, which is 870. If the check had been $1\frac{1}{2}$ low, we should have added $1\frac{1}{2}$, making 1,007 in 1,154, and the fineness would have been .873.

GENERAL REMARKS ON THE HUMID ASSAY.—Silver bullion from retorted amalgam is said to sometimes contain mercury. The presence of mercury in sensible quantity impairs the accuracy of the humid assay as described. To overcome this difficulty, add to the dissolved metal in the bottle 10 grammes (154.3 grains) of sodium acetate, and then proceed as usual. Mercury may be detected in the assay by the silver chloride remaining white when exposed to the daylight; in the absence of mercury it quickly assumes a purplish hue, finally becoming almost black. If the presence of mer-

cury is thus detected in an assay, a new assay must be made, adding the sodium acetate before the salt. If the bullion is suspected of containing mercury, the acetate may be used, as it will do no harm in any case; or, a little of the bullion may be dissolved in nitric acid, the silver precipitated by salt, and the precipitate exposed to the light. Direct sunlight acts very quickly. If the precipitate becomes discolored, it contains no mercury.

If any difficulty is found in obtaining a clear solution of the assay in the bottle, it may be boiled with nitric acid in a matrass, and afterwards transferred to the assay bottle; the matrass must be rinsed thrice with a little distilled water, and that added to the solution in the bottle. An addition of sulphuric acid is sometimes useful. The acid must be pure.

If a number of assays are to be made, they may all be dissolved at once, and each bottle dosed with 1,000 of salt, shaken down, and then a point of salt poured into each, and so on, saving time.* When the assays are finished, the bottles should be washed thrice with pure water. Care must be taken, especially in warm weather, not to allow the perspiration to come in contact with the liquids or with the interior of the vessels, because it contains salt. Salt in the water used for making the normal solution is not injurious, but if there is much, it may be allowed for. The decime salt solution should be made with pure water. The nitric acid used must not show the slightest turbidity when pure

*Sets of bottles, with pointed stoppers, may be bought at the dealers'. Also an apparatus mounted on springs for shaking a number at once.

silver is dissolved in it, nor on the addition of salt, so that the regenerated parting acid hereafter mentioned is not suitable for the humid assay. That sold in bottles marked C. P. (chemically pure) requires to be diluted.

In large establishments, the normal solution is kept in an elevated tank, from which a fixed pipette is filled by a syphon. For a description of various apparatus and methods, the reader is referred to "Mitchell's Manual," and to "Rickett's Notes on Assaying."

It is not essential that the normal pipette shall contain 100 c. c. It may be much smaller, and the solution proportionately stronger, if the neck is narrowed in similar ratio so that the measurement may be more exact. The writer has made and used pipettes of which that for the normal solution was formed from the barrel of a small glass syringe, the original nozzle being utilized as the neck, on which the mark was made by filing, and a delivery jet formed on the other end by the aid of the blowpipe. It contained less than 10 c. c., or about 145 grains of water. The auxiliary pipettes were made from glass tubes, with neck and beak like the normal. (The decime solution pipettes are usually made with a beak, but not a neck.) These were of one-tenth the capacity of the normal, instead of $\frac{1}{100}$, as is usual (but which would have been inconveniently small in this case), and to compensate this, centime, instead of decime solutions were used. The orifices were all very narrow, and the pipettes were charged by sucking, and emptied by blowing. The larger pipette not corresponding to any standard measure, it was filled to the mark with water, and emptied into a graduated cylinder as many

times as was necessary to make a definite measure, which afforded a basis for making any required quantity of a normal solution of which each pipetteful should contain 1,000 of salt. To test the accuracy of this apparatus, four separate pipettefuls of water were weighed on a delicate balance; they agreed two and two, the difference between the pairs being $\frac{1}{200}$ grain, which was closer than was necessary. Crucial test assays, the one finished with salt, the other with silver, agreed within a half point. This apparatus is far better to work with by hand than the larger one. It is necessary to add some water to the assay to furnish the requisite volume of liquid for the settling. A 10 c. c. pipette can be bought for a trifle, and the small ones will be the same as those ordinarily used if centime instead of decime solutions are employed.

The researches of G. J. Mulder have shown that, in the humid assay as described, a point may be reached at which *either* salt or silver will give a precipitate, and it has been asserted that, in a warm climate, this may give rise to an error of several points; there is, however, no danger if matters are so managed as that the check and the bullion assay are both finished in the same manner, preferably with salt, which gives the better clearance of the liquid.

RECOVERY OF SILVER.—The assays being finished, the bottles are emptied into an earthen jar, and when a sufficient quantity of silver chloride has accumulated, the liquid is poured off. The chloride is washed, and some pieces of iron or zinc and very dilute sulphuric acid are added. In a few days the silver is reduced to the metallic state in the form of a gray powder. This is washed,

dried, and melted in a black-lead crucible with some borax. As this silver will contain all the gold which was in the assays, it is not suitable for the purpose of inquartation, nor for humid assay checks.

PREPARATION OF PURE SILVER.--Dissolve the thoroughly washed chloride from the humid assays, or from the parting acid treated with salt or hydrochloric acid, in warm strong brine of common salt. Filter the solution into a porcelain or glass dish (a wash basin will serve). In the middle of the dish place a smaller porous vessel containing salt brine, with a very little nitric acid. (For a porous vessel a hessian crucible, which has stood full of water until soaked through, will answer.) Cut a piece of sheet copper of such a size as to form a cylinder which can be placed in the porous vessel, not touching the side. From one edge of the copper cut a narrow strip, but not entirely off, leaving it connected to the sheet. Take a strip of test silver long enough to form a ring around, but not touching the porous vessel, and from the edge cut a strip partly off, as with the copper. Clean the copper by immersion for a few minutes in dilute nitric acid and then in water. Connect the copper and the silver by soldering the ends of the strips, or by folding them together. Bend the plates suitably and place the copper in the porous vessel, the silver in the basin containing the solution of silver chloride, the connecting strips forming an arch above the liquids. Keep the whole warm, and, if necessary, renew the solution in the porous vessel, or add a little acid from time to time.

Pure silver in the form of small white crystals will be deposited on the silver plate. Iron or zinc in the porous

cell would work faster than copper, but would not give pure silver, as they would cause the precipitation of any trace of lead, copper, etc. which might be present, as well as of the silver. If pure silver for a plate cannot be had, a piece of coke may be used instead, being connected with the copper by means of a copper wire soldered to that, and wound around a notch cut in the coke above the liquid.

The precipitated silver should be washed, first with hot water containing some salt and a *little* hydrochloric acid, and then with pure hot water until that gives no precipitate on addition of solution of silver nitrate. If destined to be melted, the silver may be dried by evaporation after draining; otherwise it should be first pressed between sheets of filter paper, and then dried by heat, and kept in a stoppered bottle.

The dried silver may be melted in a clean black-lead pot, or in a clay crucible lined with charcoal, a little borax being added, and cast into narrow bars like those of solder. The bars may be cut into chips by the chisel, or laminated by the rolling mill, after being reduced to a thickness of about $\frac{1}{4}$ inch by hammer and anvil, being cut into suitable lengths, and annealed whenever necessary for the rolling. The filter, through which the solution of silver chloride in brine was passed, may be dried, dressed with litharge and borax, smelted in a crucible, and the resulting lead button cupelled for recovery of gold. To line a crucible with charcoal, beat in, little by little, finely ground and slightly moistened charcoal until the pot is filled, then scoop out the central portion, leaving a lining about half an inch thick, more or less. Smooth the interior by means of a glass rod or a test-tube.

RECOVERY OF ACID.—The nitric acid used in the humid assays is so far saturated with silver that its recovery would scarcely be profitable, especially as it is contaminated by sodium nitrate, resulting from the salt used in the assay. That which has been used for parting, however, contains but little silver in proportion, yet it is usually wasted, because salt is used to precipitate the silver for recovery of that, and the acid is rendered unfit for further use in assaying. The acid, as well as the silver, may be recovered in a suitable condition for reuse, the one for parting, the other for inquartation, by proceeding as follows:—

After standing undisturbed for several days, to allow any suspended gold to settle, the greater part of the acid is carefully poured or syphoned into a suitable vessel of glass or porcelain, all sediment being left behind. Hydrochloric acid is then added, little by little, to precipitate the silver as chloride, with great care not to use an excess. The liquid is briskly stirred by means of a glass rod, and then allowed to remain undisturbed until the silver chloride has settled; or, a little of it may be taken in a bottle, cleared by shaking as in the humid assay, and tested with a drop of hydrochloric acid. It is not best to attempt the *complete* precipitation of the silver, as a small quantity remaining in solution will not be injurious, while an excess of hydrochloric acid will; should the latter be accidentally added, the error can be rectified by means of some silver solution, for which purpose a portion of the original should have been set aside. When a drop of hydrochloric acid produces only a very slight cloud, the liquid is allowed to stand undisturbed for 24 hours, covered to exclude light and dust. It is

then filtered through fibrous asbestos, ground bottle glass, or quartz, until it is perfectly bright. It should now give a slight, yet distinct, precipitate on addition of dilute hydrochloric acid to a little in a test-tube, but none with solution of silver nitrate. The hydrochloric acid produces nothing but water and silver chloride, and the recovered acid is as good as any for the purpose of parting, but should not be used for the humid assay, because a trace of silver is left in it as a safeguard against the presence of chlorine. The presence of a very little copper resulting from previous partings and not removed by this treatment does no harm.

This method was introduced by the writer in 1864, in an office where a large business was done in gold bullion. It was used for years with perfect success and considerable economy. Ordinary commercial nitric acid, costing about one-tenth as much as the c. p. but often containing a little chlorine, may be rectified by the addition of a slight excess of silver nitrate, and used with perfect safety for parting.

The silver chloride precipitated from the parting acid is treated as described under *Recovery of Silver*, and is again used for inquartation of gold assays. It must contain no gold, and will contain none if the preceding directions are followed.

CONCLUSION.

ASSAYERS of bullion who work for custom must remember that they are responsible for the value of a bar as stamped by them. If any U. S. Mint, or any private

office in good standing, finds the value to be less, a *reclamation* is made by, or on behalf of, the buyer of the bar, which the assayer on whose stamp it was bought must pay if he wishes his stamp to pass in the market. It matters not that his assay may have been correct; the purchaser knows nothing of that. If the chips have been preserved, as they should be, it may, in some cases, be possible to bring the fault home to the office in which it was committed, and the first assayer, if right, may recover his money; but this will hardly avail with the mint, perhaps because "the King can do no wrong." The system pursued in the mints is such as to render mistakes almost, though not quite, impossible. An instance is within the knowledge of the writer in which a reclamation was made on a mint certificate. The error was proved to have been made in the mint, beyond reasonable doubt; but the private assayer had to suffer the loss. It is needless to say that if a bar is stamped too low, no complaint is usually made by the purchaser, though the fact, if known to the seller, might lead to a reclamation on his part, and would certainly injure the reputation of the assayer. The inference to be drawn from these remarks is obvious.

PART THREE.

PART III.

MANIPULATION, ETC.

A FILTER must be circular in order that, when placed in a funnel, the edge may be even. The edge must not extend beyond that of the funnel.

To cut a filter to fit a funnel, fold the paper twice in diametrically opposite directions; place it on a table; put the point of a pair of pencil compasses close to, not on, the corner: With a little less than the diameter of the funnel between the points of the compasses, describe an arc of a circle on the folded paper. Cut with scissors to the mark. Or, invert the funnel upon the folded paper, the rim at the corner; look down through the stem and see the edge of the paper passing through the center of the funnel. Make a mark on the paper where the rim of the funnel intersects the edge. Do likewise on the other edge and again in the middle and at as many points as may be chosen, marking the paper at a distance from the corner equal to the funnel's diameter. Cut a little within the marks. Or, fold the paper three or four times; cut straight across at a distance from the corner equal to the diameter of the required filter, then trim to an arc of a circle. Open the twice folded paper so that it shall form a cone having one thickness on the one side and three on the other.

Place the cone in a funnel; hold it with the thumb inside, and the fingers outside, and wet the paper by a jet from the wash-bottle. The filter should fit closely to the funnel all over except at the point, but especially at the rim, which should be air-tight. Press the folded edges with the rounded end of a glass rod, a test-tube, or the thumb nail.

The size of a filter should be adapted to the bulk of the precipitate when that is wanted, but when a large volume of liquid is to be filtered simply to clear it, a large filter is the more suitable. Filters already cut to a circle and of various sizes and qualities can be bought. For solubility assays of roasted ore, and wet copper assays, gray paper answers and works fast; for lead and zinc assays, a finer quality is more suitable.

In order that a filter may fit properly, the outline of the funnel, except the neck, viewed sideways must form a triangle with three equal straight sides. The worst funnel for this purpose is one that is vase shaped; the side and the neck should form an obtuse but clearly defined angle, not a curve.

In filtering, fill the filter often, not waiting for it to drain. In washing the substance on the filter, and the filter itself, let it drain before each fresh addition of water.* If the quantity of the washings is immaterial, it is best to quite fill the filter; otherwise, direct the stream from the wash-bottle onto all parts in succession, especially around the rim, which should never be found stained with the filtered liquid when dried. The matter on the filter may generally be stirred up by the stream, with advantage

*There are some exceptions to this, as in filtering and washing some sulphides which must be kept covered to prevent access of air.

to the washing. A bulky gelatinous precipitate may be carefully stirred with the rounded end of a glass rod. At last, if the precipitate is to be used, wash it down into the point of the filter. Hereafter, to save time and labor, a precipitate will often be designated by the sign p. p.

It is generally best to let a p. p. settle before filtering; then filter the liquid first, as far as practicable. In some cases water is added to the p. p., stirred, and sometimes boiled and again settled and filtered off, even several times. Finally, allow the p. p. to pass onto the filter, washing it cleanly from the vessel by a jet of water, aided if necessary by a brush formed of a clipped feather. As a general rule, in pouring from a vessel hold a glass rod to the lip, and let the fluid run down it and against the side of the filter or other receiver.

The liquid from the filter (*filtrate*) must not be allowed to fall splashing into the receiving vessel, but should trickle down its side. Even if the filtrate is not required, a neat workman will not have it splashed about the table.

There are several ways of ascertaining when the washing of a p. p. is completed. If the filtrate was acid, the washings must cease to redden blued litmus paper; if alkaline, they must not discharge the color of reddened litmus paper. If the water used be pure, a drop from the filter, evaporated on a piece of glass, should leave no film, or only a slight speck.

To dry a p. p. first let it be well drained, which may be aided by removing the triple side of the filter from contact with the funnel, and pressing it slightly against the opposite side, or in some cases, by sucking the stem of the funnel, but not too forcibly lest the paper be ruptured.

The filter, with the p. p., may be carefully removed from the funnel, unfolded, and laid on a warm surface, better with several thicknesses of paper under it. Special absorbent tiles are also used. Or the filter with its contents may be dried in the funnel, that being placed in the upper end of a conical tube standing on a sand-bath. The drying should be continued until two weighings after thorough cooling have given the same result.

To remove a dried p. p. from a filter, invert that on a sheet of glazed paper, and manipulate it with the fingers to loosen the p. p. Scrape it with a wooden or horn spatula; double it and rub the surfaces together; lay it flat and brush it with a rather hard brush, etc., according to circumstances.

To use a Mohr burette (see wet copper assays), fix it in a vertical position. In reading it, if there is no float, the middle of the concave surface of the liquid is the indicator. The eye must be level with the mark. With a float, the mark around that is the indicator, and the eye must be level with it, so that it appears as a line, not an oval ring. In charging the burette, first fill it, and see that no air bubbles remain; then, by compressing the pinch-cock, which opens it, allow the liquid to escape into a vessel beneath, until all air is expelled from the neck, and then refill and again let the liquid flow until the indicator is exactly coincident with the upper mark. It is then ready for use.

A draught place should be provided in which acids may be boiled and hydrogen sulphide used while the fumes are conducted away by a flue. Such a closet is conveniently made by fitting a box on the outer side of

the lower half of a window, so that the lower sash forms a front which can be opened or closed, and through which the progress of the work may be observed. It is still better if the back be glazed also. In the top or roof of the closet is a small wooden flue which, if convenient, may be connected with a chimney. Within the closet the coal-oil stove, lamps, etc., are placed.

Hydrogen sulphide is a gas which has the odor of rotten eggs. It is produced by the action of dilute sulphuric acid on iron protosulphide, which may be made by melting iron filings or borings with sulphur, or may be bought. The gas is conducted, by rubber and glass tubing, into water to wash it, and then into the beaker or other vessel containing the liquid treated. It is used in certain wet assays to precipitate certain metals from solution. Various kinds of apparatus for its production can be bought. For all the purposes of this book an eight-ounce generator will suffice.

Wash all vessels after use. Balls of wet paper are useful for cleaning the interior of matrasses, etc. Test tubes sometimes require a swab on a slip of wood, or a test-tube brush. To rinse a matrass, flask, bottle, or tube, put some water in it, stop the mouth with the thumb, invert the vessel, shake it so as to wash all matter down to the mouth; then remove the thumb. Warm water with a little hydrochloric acid will remove iron oxide and many other residues. Vessels which are to be used for parting, for the humid assay of silver, or for any other purpose in which a trace of a chloride or chlorine would be objectionable, must be rinsed three times with pure water. Vessels which have been used in the new

copper assay, etc., with potassium zanthate, are best cleaned by a solution of potassium cyanide followed by water. The clippings of filter paper are useful for drying glassware.

The motto of a work-shop should be, "*A job is not finished until everything is put in order.*"

NOTE.—Becker's assay balance No. 5 is strong enough for the weighing of most of the wet assays, while also delicate enough for that of beads from gold and silver ores. Lead and copper, etc., buttons may be weighed on the pulp scales.

LEAD ORES.

FIRE ASSAY.—All lead ores can be assayed by melting with soda, borax, iron, and carbon. There is always a loss of lead in the fire assay. Many of our lead ores contain copper and zinc, and if sulphur is absent, the copper, or some of it, will be got with the lead, and zinc will largely increase the loss.

It is therefore best, for those not well acquainted with the different ores, to convert all kinds into one class, to a certain extent, by an addition of sulphur, unless it is evident that sulphur is present in sufficient quantity to combine with all the copper and zinc which the ore may contain. The following prescription, slightly varied as to the proportions of soda and borax, and in some cases with an addition of glass, to suit the different gangues (see Part I, Article "Materials") will give results sufficiently accurate for many purposes of metallurgy and prospecting. The ore should be passed through a sieve of 40 meshes to the running inch.

Ore.....	1 part
Soda.....	1 "
Dried Borax.....	1 "
Flour.....	$\frac{1}{5}$ "
Sulphur....	$\frac{1}{5}$ "

The sulphur may be omitted if the ore contains no

metallic nor oxidized copper, as blue or green carbonate, black or red oxide, and no zinc except in the form of blende; also when the ore contains any considerable quantity of sulphur in the form of galena or pyrites, but zinc blende forms no exception, requiring all its own sulphur. Mix and place in a crucible; cover with salt, and some more borax. Melt with *low red heat*. Stir with the heated end of a piece of nail-rod as long as that melts away rapidly; then, from the same end of the rod, cut a piece of a length to suit the pot and put the piece into the pot, together with any lead or slag that may have adhered to it. Cover and heat to rather bright redness for ten minutes. Remove the remnant of iron, after washing it in the slag (see Part I, page 61); shake and tap as in the assay of gold and silver ores, and either pour into the assay mould, or, which is better in the case of poor ores, cool and break the pot.

If the button is coppery, not enough sulphur was present; if hard or brittle, the assay probably required a longer time in the furnace, or it contains antimony, in which case it will be better to repeat the assay, using more soda and omitting the iron and flour, also the sulphur if the ore is antimonial galena, but if the ore is a thoroughly oxidized one the sulphur may be increased. Clean it by hammering and brushing. If the ore contained arsenic, the button will consist of two distinct parts, the one hard and brittle, which is iron arsenide, to be rejected, the other malleable, which is lead. They can be separated readily.*

If 100 grains of ore were taken for the assay, the

*See also Appendix to Part I.

weight of the button in grains is the percentage; if 10 grammes, the button weight in grammes multiplied by 10. An addition of 5 per cent. of the button weight may be allowed for loss.

Carbonate ores, free from copper and zinc, require only to be melted with soda, borax and flour, but it is as well to add a nail or piece of thick wire, in case there may be some sulphate or arsenate also. (A mixture of carbonate and sulphate, in the right proportions will yield all the lead without iron or carbon, but this cannot be depended on when the proportions are unknown, and arsenic in any form calls for iron.) The button may be cupelled for precious metal, and, if worth notice, the weight of that found deducted.

WET ASSAY.—This is more accurate than any fire assay for lead.

Treat a suitable quantity of the very finely powdered ore with strong nitric acid in a porcelain dish, using as a cover an inverted funnel of such a size as to rest *within* the rim of the dish; or in a casserole covered with a clock-glass, concave side upward. Boil gently on the sand-bath or coal-oil stove until the residue is as nearly white as it can be made, and the most of the acid has evaporated. Cool, and dilute with water. Add dilute sulphuric acid as long as it makes a precipitate; not much more, as a large quantity may throw down a p. p. of lime where only lead is wanted. Remove the cover, and wash into the dish any matter that may be on it. Filter, and wash with water slightly soured with sulphuric acid, until the drippings no longer produce more than a faint *white* cloud in a little solution of sodium carbonate.

Pierce the filter with a glass rod, and wash the p. p. off it into a porcelain dish; then carefully remove the filter from the funnel, unfold it, and wash into the dish any portion of p. p. which may have lodged in the folds. Use no more water than is necessary, and if, nevertheless, the quantity is large, let the p. p. settle, and pour off a part of the water. Add as much sodium carbonate as will dissolve, and boil gently for half an hour, keeping the vessel covered, as before, by an inverted funnel or a glass. Add a little more sodium carbonate, and observe if it causes effervescence. It will not if enough has been used.* Filter, and wash with water until the drippings no longer make a precipitate in solution of barium chloride, nor in one of copper sulphate. The lead is now on the filter in the form of carbonate.

Place a clean beaker under the filter; again pierce that, and wash it clean. Redissolve the lead carbonate by dilute nitric acid, or by acetic acid†. Add dilute sulphuric acid freely. Allow the p. p. to settle; filter; wash with water soured with sulphuric acid, and lastly, once with pure water. The lead is now again on the filter in the form of sulphate, as it was after the first filtration, but freed from impurity.

Dry the p. p. on the filter; transfer it to a small porcelain basin; heat nearly to redness; cool and weigh. If very particular, burn the filter in another basin until the

*Sodium bicarbonate may be used in the first place, but will not answer for this test. The potassium salts may be used in place of the corresponding sodium salts.

†The lead carbonate may be dissolved on the filter without previous removal.

ashes are white; moisten with nitric acid; add a drop of sulphuric acid; dry, and add to the lead sulphate in the first basin before heating. To be still more exact, burn another filter of the same size, treat the ashes in the same way, and put them on the weight pan when weighing. The weight of the lead sulphate multiplied by 0.6833 gives the weight of the lead.

Instead of heating and weighing the p. p., it may be mixed with about twice its weight of potassium cyanide, placed in a small crucible, covered with salt, melted at as low heat as possible, cooled, extracted by breaking the pot, and weighed as pure lead.

Again, and better if the ore contain much lime, acetic acid being used for the dissolving of the lead carbonate, the lead may be precipitated as chromate by solution of potassium bichromate, collected on a filter, dried on a water-bath, and weighed, filter and all, another filter of the same weight being dried at the same time and placed on the weight pan of the scales. Calculate 68.94 per cent. of the weight as lead.

Time may be saved by proceeding as follows: Instead of boiling the first residue with sodium carbonate, digest it, cold, with a mixture of ammonia and acetic acid in which the acid is in slight excess, or with solution of ammonium acetate to which a little acetic acid has been added. Filter and wash. To the filtrate, including the washings, add diluted sulphuric acid as long as it causes a p. p. Again filter; wash with soured water; dry, calcine and weigh. The lead is in the form of sulphate, as in the first process.

Unless for particular work, the lead sulphate may be

weighed on the filter after drying at 248° Fahrenheit, a similar filter, similarly dried being placed on the weight pan.

Lime is the chief common constituent of lead ores which is likely to cause an erroneous result. If calcium sulphate (sulphate of lime) is allowed to remain with the lead sulphate at the first filtration, it will also be converted into carbonate by the boiling with sodium carbonate, or it, as well as the lead sulphate, will be dissolved by ammonium acetate. In either case it is liable to be thrown down again, together with the lead, as sulphate in the last precipitation. But, while lead sulphate is *very* little soluble in water, especially if the water contains sulphuric acid, calcium sulphate is considerably so. It is for these reasons that stress is laid on the use of quite dilute acid for the precipitation, and of acid water for the washing. It has already been shown that the lead may be precipitated from the acetic acid solution as chromate, any lime present then remaining in solution and passing off in the filtrate. I also find that an acid solution of ammonium acetate saturated with lead sulphate can still dissolve calcium sulphate freely; hence the latter may be removed from the former by washing with such a saturated solution, after which that must be washed out by water.

COPPER ORES.

THE fire assay of copper is not very satisfactory, especially in unpracticed hands; it may, however, answer the purpose of a prospector. The ore must be ground to pass a 40-mesh sieve. Poor ores, and all which contain lime or baryta and sulphur in any form, should be submitted to a matte smelting; the first in order to obtain an enriched product on which to operate; the second in order to remove calcium or barium sulphate which would cause a loss of copper. It is well to add a large nail to a matte smelting to separate any lead which may be present.

ORES CONTAINING LESS THAN 8 PER CENT. OF COPPER.

Ore.....	400 grains.
Borax glass.....	400 “
Flour.....	20 “
Sulphur.....	10 “

Mix by grinding in wedgewood mortar; put in pot, cover with salt, and over that put 400 grains more borax glass. The sulphur and flour may be omitted if the ore contains sulphurets, zinc-blende excepted, provided none of the higher metal-oxides are present. If there is any suspicion of lead, add a nail. Melt slowly and finish with strong heat. Remove the nail; cool and break the pot. Separate the matte from the overlying slag and from lead, if any, and reserve for further treatment.

RICHER ORES WITH LIME OR BARYTA GANGUE AND CONTAINING SULPHUR EITHER AS SULPHURETS OF ANY KIND, OR COMBINED WITH THE GANGUE AS GYPSUM OR HEAVY SPAR.*

Ore.....100 grains.

Borax glass.....100 “

Sulphur if required.

Mix, put in pot, cover with salt and more borax, and add a nail if lead is suspected. Melt, etc., as before. Reserve the matte.

RICH SULPHURETTED ORES WITHOUT LEAD, LIME, OR BARYTA.—These include the mattes from the preceding as well as natural ores. Pulverize; roast in a roasting dish in the muffle, beginning with a low heat, and raising it gradually to bright redness, stirring the ore at intervals with a piece of wire. If the ore is inclined to decrepitate and fly out of the dish, it must be covered by another roasting dish inverted and having some notches in the edge and a hole in the middle, and must be cooled before being uncovered for the stirring. It will soon cease decrepitating. When no more odor is perceptible from the red-hot ore, cool, grind again with the addition of one-tenth part of coke. Roast again, observing whether arsenic is present or not. Arsenic is known by an odor like that of garlic. Roast until all the coke is burned to ashes. If arsenic was observed, repeat the grinding with coke and roasting until it is all expelled. At last place on the ore a piece of ammonium carbonate of 10 or 20 grains weight, cover and heat until the carbonate has evaporated; this removes any lingering sulphur. The ore is now in the following class.

* In this and preceding, strontia may be regarded in the same light as baryta.

RICH OXIDIZED ORES.—If the ore is a natural oxide, the assay must be weighed and calcined by heating to redness in a roasting dish. Add to the assay—

Soda an equal weight.

Argol “

Lime one-fourth the weight.

And, if it contains much quartz, a little powdered iron-stone (hematite) or forge scale. Mix well in the wedge-wood mortar. Place in a pot which should not be more than two-thirds filled. Cover with a mixture of soda and argol in equal parts, and on that a weight of borax glass equal to the weight of the assay. Heat very slowly to dull redness for fifteen minutes, then fuse thoroughly with high heat, and remove from the fire without delay. Wash the side of the pot by the slag as in an assay for gold and silver, tap to settle all globules, allow to cool, and break the pot.

The copper should be found at the bottom of the pot in a single button. If the button is tolerably malleable, and when broken or cracked is found to be of a good red color all through, it is pure enough, and may be weighed. If it is black, gray, or mottled, it must be refined by cupellation. The slag should be neither red nor green. The side of the pot should be but little colored with red or green.

REFINING THE IMPURE COPPER.—Place the button on a highly heated cupel, and an equal weight of pure copper on another. Add to each one-tenth part of lead, and close the muffle after placing some glowing coals in it. When the buttons are well melted, open the muffle

to allow of the entrance of air; the refining then begins. The button behaves much in the same manner as one of silver, when nearly free from lead, but the final brightening is less vivid. The heat must be higher than for silver, and should be such as that the copper solidifies at the instant when the lead leaves it. The button should then appear greenish. If it does not seem to be pure, add another tenth of lead, both to the assay and to the pure copper beside it. Continue thus adding lead by tenths and cupelling it off, until the assay button is pure. Remove from the muffle and plunge both of the buttons into cold water as soon as possible after solidification in order to stop oxidation.* Clean by hammering and brushing; weigh, and to the assay button weight add as much as the pure copper has lost. If pure copper is not at hand, refine the assay button and weigh it; then cupel it again with as many tenths of lead as were used in the refining; weigh again, and to the first weight add as much as has been lost in the second cupellation. The percentage must be calculated on the quantity of ore originally taken, whether matte smelted or not.

Argol is crude cream of tartar; an intimate mixture of one part of flour and four of soda may be used instead.

WET ASSAY.—Unless with very pure oxidized ores, the wet method is less troublesome, as well as more exact than a fire assay. A suitable quantity of the finely powdered ore is gently boiled with a mixture of nitric and hydrochloric acid, more of the first than of the last,

*Some operators cover the button with charcoal powder at the instant of brightening, with the same view.

until decomposed, the sulphur, if any be present, either being completely oxidized into sulphuric acid, or melted to clear amber colored drops.

It is best that the sulphur be oxidized. The oxidation is promoted by adding, from time to time, a few crystals of potassium chlorate. As the solution is to be evaporated, it is best made in a casserole covered by a clock-glass, or in a dish covered by a funnel which fits inside of the dish. It may be made in a matrass, which is very suitable for the boiling, but then it must be transferred to a dish or other open vessel for the evaporation, or time must be lost. If the sulphur is not dissolved by oxidation, the assay must be cooled until the drops of sulphur become solid so that they may be removed to a small dish and burned. Any residue is then to be dissolved in hydrochloric acid and added to the main solution.

The solution must now be evaporated to dryness after the addition of sulphuric acid to insure the expulsion of all nitric acid. Dry with gentle heat, not causing the liquid to bubble and spirt; toward the last it may be necessary to stir the thickening mass in order to prevent spirting. Allow to cool; add a little water and a few drops of hydrochloric or sulphuric acid. Allow to remain half an hour in a warm place, and stir once or twice with a glass rod. Filter and wash, receiving the liquid in a clean beaker. To the filtrate and washings in the beaker add ammonia in excess, that is until the liquid smells strongly of it.

If a considerable brown flocculent p. p. forms on the addition of ammonia, any arsenic which may be present

will go with that; otherwise add "magnesia mixture" as long as it produces a turbidity. Let the assay stand as long as is convenient, in a warm place, then filter and wash with water containing ammonia as long as the drippings have a blue tint. To the filtrate add hydrochloric or sulphuric acid until it is quite acid; warm slightly, and place in the beaker a piece of clean sheet iron of such a size that, while completely covered by the liquid, it does not lie flat on the bottom of the vessel.

Heat the liquid gradually to almost boiling. Bubbles of hydrogen gas will be seen rising from the surface of the iron, and this action must be kept up, in moderation, by adding hydrochloric or sulphuric acid from time to time. When the liquid is quite colorless, test it by holding a clean wire or other piece of iron in it for half a minute; if the wire is not discolored no copper remains in the solution. A piece of zinc is even better than iron for the test, and zinc may be used in place of iron for the precipitation of the copper if it is certain that no nitric acid nor nitrates remained after the evaporation with sulphuric acid. The beaker should be covered during the precipitation of the copper, to exclude dust.

The copper is now in the metallic state, precipitated by the iron a corresponding portion of which has been dissolved. Pour off a great part of the liquid, with care that no copper goes with it. Transfer the remaining contents of the beaker to a dish*, and detach the copper from the iron by means of a clipped feather. Reject

*This is best done by filling the beaker with water, inverting the dish over it, and operating in a manner similar to that described for cupping a gold assay, but the beaker must be removed by sliding it quickly to one side.

the iron and wash the copper six times with hot pure water, draining it off as closely as possible each time into another dish in which any small particles of copper that may unavoidably escape may be settled and eventually recovered. The last two washings are best made with alcohol, but it is not essential. The spongy copper may be compacted by pressing with the finger, or the bottom of a test-tube, to aid the washing and draining. Some light black particles of carbon from the iron will probably be seen; these should be washed away as much as possible. A slight discoloration of the precipitated copper may often be removed by gentle boiling in dilute hydrochloric acid. The proper color is a pure copper red.

The washed and drained metal is to be dried in the dish on the water-bath. The percentage is calculated for the weight of ore used, which may vary, with the supposed richness and the delicacy of the balance, from 20 grains to several hundred.

Russia sheet iron is suitable for the precipitation of copper if the scale is removed by means of warm dilute hydrochloric or sulphuric acid and rubbing. Good wire cut to a suitable length is also proper; it may be cleaned with sand-paper. If zinc is used, it should be such as dissolves without residue in dilute hydrochloric acid.

Magnesia mixture, for the removal of arsenic in the case of an ore containing little or no iron, is prepared by dissolving epsom salts in water and adding, first sal ammoniac (ammonium chloride), and then ammonia. The ammonia should produce no p. p., and it will not if enough sal ammoniac be present.

The dried copper should retain a good color, and should

not adhere to the dish in which it was dried, and it will not if well washed. The result is more reliable if the dried copper is calcined at a red heat in the muffle, cooled, moistened with nitric acid, again heated to redness, cooled and weighed. It is then in the form of black oxide, of which 79.85 per cent. is copper.

An oxidized ore, whether a natural product or the result of roasting a sulphuret, if free from arsenic, antimony, and bismuth, requires only to be decomposed by hydrochloric and sulphuric acid, the solution filtered, and the copper precipitated by iron or zinc, etc. The red oxide, however, requires an addition of potassium chlorate to the solution in quantity sufficient to convert that from a brown and muddy appearance to a clear green, after which it must be boiled for some time to expel excess of chlorine before the precipitation is attempted. Nitric acid will answer as well as potassium chlorate, only then the solution must be evaporated to dryness as in the first described process. Ores which do not yield all their copper to acids may be made to do so by previous fusion with soda in a porcelain crucible.

A better way in which to precipitate the copper is to pour the solution into a platinum dish of known weight, and add a piece of pure zinc. The copper is precipitated on the dish, to which it adheres, making the washing easy. The whole of the zinc is to be dissolved, acid being added, if necessary, for that purpose until no more bubbles are seen. The copper is dried and weighed still on the dish, the known weight of the latter deducted, leaving the net weight of the copper. The copper is removed from the dish by nitric acid, which does not attack the dish.

But little inferior is the method of precipitating the copper in a porcelain or glass vessel in which is placed a weighed piece of platinum foil, and, in contact with that, a piece of zinc. The copper is precipitated on the platinum. In these processes, all nitric and hydrochloric acid is expelled from the solution by boiling down with sulphuric acid.

The method which is generally considered the best at present is the precipitation by electrolysis, in which a current of electricity is passed through the solution by means of platinum electrodes, on one of which the copper is deposited in a compact form. The nitric solution is used in this process, hydrochloric acid being injurious. The method requires special apparatus and experience, and though very exact is equaled in this respect and surpassed in point of expedition by my new process, to be described shortly.

VOLUMETRIC ASSAYS OF COPPER.

THERE are a great many methods of estimating copper volumetrically, that is, by the *volume* of a reagent which is found to be necessary to produce or complete a certain effect in the suitably prepared solution. The one most commonly used depends on the fact that the blue ammoniacal solution of copper is decolorized by a solution of potassium cyanide. This is generally known as

PARKES' PROCESS.—Take 10 grains, or 1 gramme, of pure copper: dissolve it in nitric acid; add ammonia until the p. p. at first formed is redissolved to a blue liquid; add water to make about one-third of a pint (in a beaker). Charge a Mohr burette with a solution of potassium cyanide made by dissolving one troy ounce in five and a half fluid ounces of water (or the water may be weighed) and filtering if necessary. Run the liquid from the burette, into the blue solution of copper, stirring that with a glass rod, until the blue color becomes faint and purplish; continue cautiously, adding the cyanide little by little, finally drop by drop with intervals between, until only a faint tint remains which disappears in a few minutes, leaving the liquid colorless. Observe how many c. c. of the cyanide have been used, by the marks on the burette, and note the number. The cyanide solution is thus *titrated*, that is, it is known what volume of it corresponds to 10 grains or to 1 gramme of copper in the

solution. The titrated cyanide solution should be kept in a dark-colored stoppered bottle.

To assay an ore: take 20 grains or 2 grammes, more or less according to the grade of richness, and make an acid solution by one of the methods previously described, or by nitric acid alone. Add ammonia in considerable excess, and heat moderately for some time. If there is any considerable quantity of insoluble matter, filter, and wash with water containing ammonia until that passes through colorless. The entire volume of solution, including the washings if filtered, should be about one-third of a pint. When quite cold add solution of potassium cyanide from the burette precisely as in titrating, noting the quantity used.

The determination of the assay is now a mere sum in proportion or the "rule of three." For instance, if 10 grains of pure copper required 45 c. c. of cyanide solution, and the ore assay required 63.5 c. c. then $45:10::63.5:$ the copper in the assay $=14.1$ grains, and if 20 grains of ore were taken, the percentage is $14.1 \times 5 = 70.5$ or, if 50 grains of ore were used, $14.1 \times 2 = 28.2$ per cent.

The presence of manganese interferes a little with the decoloration. This is obviated by an addition of ammonium carbonate. Arsenic also interferes but may be prevented by "magnesia mixture," added before filtering. Nickel, cobalt, or zinc is fatal to the accuracy of the process. When either of these is present, the copper should be precipitated by iron or zinc as in the first described process, washed, and, without drying, dissolved in nitric acid, blued by ammonia and determined as above.

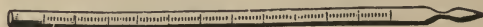
The process is rapid and easy, therefore popular, but not always accurate. The quantity of ammonia and of

the different acids used, affects the result. On account of variations of temperature, a check test on pure copper should be made with each set of assays unless the temperature is nearly constant. This applies however to all volumetric processes. The cyanide solution is liable to change, despite the precaution of keeping it as much as possible in darkness, hence too large a quantity should not be made at one time; the directions given above for making it only indicate approximately the proportions to be used, which may be modified to suit convenience, and the quantity prepared may be adapted to the expected demand.



Gay Lussac
Burette.

The cuts show two kinds of burettes which are suitable for this and other volumetric assays. The Mohr burette is the most convenient. It may be of 50 or 100 c. c. capacity; should be graduated to one-tenth c. c. and furnished with Erdman's float. The discharge by means of rubber tube and pinch-cock is preferable, for common use, to that by a glass cock.



MOHR'S BURETTES,

WITH PINCH COCK, RUBBER TUBING AND TOP.						
Size.....	10x1-10	10x1-5	25x1-10	25x1-5	50x1-10	50x1-5
Price....	\$1.25	1.12	1.40	1.25	1.60	1.50
Size.....	100x1-5	100x1-1	200x1-1			
Price....	\$2.00	1.50	2.00			

ASSAY BY AMALGAMATION.—This may answer the purpose where means are wanting for the other methods. Take 100 grains or more of the powdered ore, and, if it contains sulphurets, roast it. Grind it in an iron mortar, warmed, with water enough to make a pulp, and such a quantity of a mixture of iron sulphate (copperas or green vitriol) and salt as may be necessary to cause a copper stain on a piece of clean iron held in the hot pulp for a few seconds; also a little mercury. Continue rubbing and stirring with the pestle until the latter no longer takes up mercury when lifted out, and a bright piece of iron is not stained by the pulp. Then wash the pulp away by a stream of water, leaving the mercury. Strain the mercury through a piece of wet buckskin by twisting. Copper, perhaps also lead, gold, and silver, will remain in the form of a ball of amalgam. Tie the ball in a small piece of cotton cloth, as a dumpling is tied; put it into a crucible; cover it with powdered charcoal; heat it gradually to redness; then add some borax and a piece of charcoal and heat strongly. When cold, break the pot in which a button of metal will be found. If the button is good copper, weigh it; if not, refine it as directed for the crucible assay, and then weigh. If the button remains of a pale color after refining, it probably contains a large proportion of silver. Treat it by scorification and cupellation for precious metal. From the net weight of copper obtained, calculate the percentage for the weight of ore taken.

NEW PROCESS.—This is the writer's own invention. It is not difficult and, in point of accuracy, is believed to be inferior to none. Copper in ammoniacal solution is

completely precipitated by solution of potassium zanthate. By this reagent copper is easily detected in a solution which is not visibly blued by addition of ammonia. Neither arsenic, nickel, zinc, manganese, nor the earths interfere. So delicate is this method that it is possible to make the assay on one decigramme of ore to one-tenth of one per cent. I prefer to operate on larger quantities when accuracy is requisite.

Weigh ten grains, or one-half gramme, of pure copper, and dissolve it in nitric acid. Add ammonia in excess, and water to make about half a pint of solution, in a beaker of thin clear glass. From a Mohr burette, which should be graduated at least to one-fifth c. c. run in solution of potassium zanthate, stirring gently the while, or at short intervals, with a glass rod. The solution in the beaker soon appears green from admixture with the yellow p. p. As long as a green color is plainly seen on stopping the flow of the zanthate, and gentle stirring, more zanthate may be freely added. When the green color grows faint, proceed with caution. Stir actively for half a minute or more, but avoid making froth. Allow the p. p. to settle to some extent, and if the liquid is not clear enough, stir again, but as long as a green tint is perceptible it is not necessary that it should be very clear. Continue adding zanthate, by quarter c. c. or less, stirring and settling. When near the end, stir and settle until the liquid is no longer turbid, although flecks of the p. p. may remain suspended. After this a very little stirring will suffice after each addition of a drop or two of zanthate. When an addition of zanthate causes no cloud in the liquid, the precipitation is complete and a slight excess of the zanthate is present.

For the purpose of verification, a second assay may be made, and nearly the quantity of zanthate required run in at once, finishing with caution as before. To verify the presence of a slight excess of zanthate, stir and settle; then draw through the clear liquid the end of a small glass rod moistened with an ammoniacal solution of copper. Either a faint cloud will appear near the surface, or minute drops of the blue solution will sink through the liquid, leaving thin white streaks behind them. The appearance of these streaks, which assume the form of wreaths from the motion of the liquid, is very beautiful.

Toward the end of the operation, a record should be kept of the quarter or tenth c. c. of zanthate added, in order that, in finally reading the burette, the last addition, which produced no p. p. and was therefore not required, and half of the one before the last, because it was probably only partially requisite, may be rejected. Thus it is known how many c. c. of the zanthate are required to precipitate the quantity of copper taken, which constitutes the *titre* of the solution (or the *standard*).

To assay an ore, take such a weight as may be supposed to contain from one to ten grains of copper, or a corresponding proportion in grammes if that system of weight is in use. Treat with a mixture of nitric, hydrochloric and sulphuric acid, boiling gently in a covered vessel or a mattrass, until decomposed and all sulphur is oxidized; cool, dilute, filter and wash. To the filtrate and washings add ammonia in excess; filter, wash with water containing ammonia until it gives no yellow color on testing a little of it in a test-tube with a glass rod moistened

with zanthate. Let the entire volume of solution be about half a pint, adding water if necessary to complete that quantity, then proceed with the zanthate as before. From the number of c. c. used, and the titre of the zanthate, calculate the quantity of copper in the assay, as in other volumetric methods, and from this quantity reckon the percentage on the weight of ore taken.

In making this assay, the zanthate should at first be run down the side of the beaker, to prevent frothing and consequent floating of the p. p.; the smaller quantities added toward the last are best allowed to fall upon the stirring rod resting in the beaker. As long as much p. p. is formed, it may be plainly seen by looking downward on the solution, but the faint clouds produced later are best seen, as in the humid assay of silver, by holding the glass toward the light, and looking obliquely upward through the liquid. The liquid clears better if there is a goodly quantity of p. p. than otherwise. It clears best when the precipitation is near completion. Excessive stirring is disadvantageous by breaking the curd; hence stir but little, and gently, until a more perfect clearing becomes necessary, then agitate briskly for a minute or two, and settle enough to leave a stratum of clear liquid at the top before adding more zanthate. After this, a very slight stirring suffices.

There are three guides to the ending: Firstly, the disappearance of the green color, and darkening of the yellow; secondly, the more perfect clearing, and thirdly, the cessation of clouding on addition of zanthate. To make the assay well some practice is required; the novice is almost sure to run in too much of the zanthate. It would

be quite feasible to have a titrated solution of copper with which to work back by adding a known quantity, and, in the final reckoning, deducting a corresponding weight from the total result. The most convenient way in which to make such a solution is to dissolve some bluestone in water, and then titrate a certain measure of the liquid, which should be quite a weak solution. To use it, measure the required quantity, add ammonia, and pour the whole into the assay; then proceed again with zanthate.

In order to be quite sure in the case of a very complex ore, boil the first acid solution, before filtering, down until heavy white fumes of sulphuric acid come off; cool; add a little water and a piece of iron; keep the liquid acid with sulphuric acid, and digest warm until the liquid no longer stains a piece of clean iron; filter, wash, redissolve the precipitated metals in mixed acids as before, add ammonia, filter and proceed with the assay. This will be necessary when an appreciable quantity of cobalt is present in the ore. The copper p. p. should be mustard yellow although, with very strong zanthate, it may appear darker at first. If it is distinctly brown, there is probably cobalt in the assay, or the zanthate may be out of order, which can be tested in a moment by means of some ammoniacal solution of copper.

After the precipitation of the copper, a very slight excess of zanthate being present, if acetic acid, added to the clear liquid in slight excess, produces a reddish yellow p. p, nickel is present; and if greenish turbidity, either at once or on further addition of zanthate, cobalt is also

present, and another portion of it will be with the copper p. p., spoiling the assay, which must then be repeated as directed above. By again adding ammonia, the nickel p. p. is redissolved and the dirty greenish cobalt p. p. remains.

PREPARATION OF THE POTASSIUM ZANTHATE SOLUTION.—Dissolve two ounces (troy) of caustic potassa in about a quarter pint of 95 per cent. alcohol in a stoppered bottle; add carbon bisulphide, little by little, as long as a curd is produced, a hissing noise heard, and a partial vacuum is formed in the bottle, as evinced by the stopper resisting extraction. Shake the bottle occasionally. When the above effects cease, and a further addition of the bisulphide causes expansion, which may expel the stopper, while its odor is no longer neutralized but becomes disagreeably perceptible, and a yellowish fluid appears which partly liquifies the previously almost solidified contents of the bottle, pour the whole into a dish and leave it to spontaneous evaporation in a dark, cool place for twelve hours or more. The dish will then contain a pinkish, plastic mass, possessed of an evil odor. Dissolve this in six pints of cold pure water. Let the solution stand a short time to deposit any excess of bisulphide, and then bottle it in common beer bottles ("black bottles") which keep in a cool, dark place.

About 50 c. c. of the solution thus made will precipitate 0.5 gramme of copper. It may be diluted, if so preferred for very close work, so that it will require 100 or 200 c. c. for that quantity of copper. If the alcohol is not almost saturated with caustic potassa, the curdy mass spoken of may not appear until evaporation has

taken place. The compound may be made in an open dish, the carbon bisulphide being dropped into the alcoholic solution of potassa until that no longer reacts alkaline, or as long as it will dissolve by stirring. The liquid is then left to evaporate.

Regnault, that prince of chemists, who has investigated almost every known compound, and whose name is constantly on the lips and pens of other chemists, says that with a solution of absolute alcohol, the greater part of the potassium zanthate falls out at once in orange colored crystals, and I have seen it so. The method I have given is that which I use, and it answers my purpose well. The solution soon decomposes in colorless bottles, even when made with boiled water, and kept with exclusion of air, or in an atmosphere of carbon bisulphide; in fact, sooner in the latter case, a turbidity appearing at the top, and gradually spreading. An addition of potassium hydrate produces a flocculent precipitate, after the settling of which the liquid remains clear, but soon becomes unfit for copper assaying. In a beer bottle it remains clear and retains its properties for a number of weeks. From these facts it is inferred that light is the chief cause of the decomposition.

ELECTROLYTIC DETERMINATION OF COPPER IN ORES, ETC.

[Contributed by Mr. A. K. Brewer, of Tucson, Arizona.]

A MOST accurate and simple determination of copper is that by precipitation from a nitric acid solution upon platinum foil, by means of the electric current. The apparatus required is a battery of one or more cells, according to amount of work done, a few platinum cylinders and spirals, some small narrow beakers about two inches high, porcelain capsules, etc. A very satisfactory type of battery is Fuller's* bichromate; it is constant as well as powerful. The chemicals used with this battery are quicksilver, bichromate of potash and sulphuric acid.

To make the platinum cylinder, upon which the copper is to be deposited, take a piece of platinum foil of medium thickness, two by two and a half inches, and with the blow-pipe solder to one corner, using a minute fragment of gold, a medium thick platinum wire three inches long. Then roll foil into a cylindrical or a spiral form of, say, half an inch in diameter and two inches long. For the other pole take a platinum wire about eight inches long, and twist three inches of it into a spiral whose outer ring

*On this coast Fuller's battery is manufactured only by the Pacific Electrical Works, Sacramento, Cal., and sold at \$2.50 per large cell.

will just fit the bottom of the beaker; the rings turning inward to the center of beaker, when the wire is bent sharply up, so that the spiral will sit in the bottom with five inches of wire extending straight up through the center of beaker. Put a narrow glass tube two inches long over the wire so that it rests on the spiral at the bottom. The object of the tube is to prevent contact between the cylinder and spiral when one encircles the other.

It will be found convenient to fasten to the work-table two parallel, horizontal brass or iron rods (thick wire will do) about fifteen inches long and three inches apart, and raised one and a half inches from the table. Between these rods are placed the beakers; in this manner five assays or more may be carried on together and any one of them can be detached without disturbing the others. I have been using four cells of Fuller's battery and can run with that power from four to six assays at a time.

The zincs and carbons are coupled alternately, and a wire from the zinc pole is attached permanently to the left hand horizontal rod; wires run up to the table from each carbon, so that, by connecting the first, second, third, or fourth carbon with the right hand rod, I can use one, two, three, or four cells, as may be desired. No more current should be used than is necessary, for a strong battery may cause zinc to precipitate on the foil.

One gramme of the finely pulverized ore, if poor in copper, or one-half gramme, if rich, is treated with nitric acid in a small flask until complete solution of the copper takes place, then excess of acid is evaporated off until residue is only just covered with acid. A few drops of sulphuric acid are added to precipitate lead, and the mass taken up

with a little water. The solution is filtered into a very small beaker and the filter washed with no more water than is necessary. The bulk of the solution is preferably not more than 20 cubic centimeters (two-thirds of an ounce). To make the solution uniform it is stirred with the spiral wire, which is then dropped into the beaker. The platinum cylinder is put over this, nearly, but not quite touching the spiral at the bottom. Connections are then made with the battery; the cylinder being joined to the zinc, or negative pole, and the spiral to the carbon, or positive. Bubbles will rise from the spiral and the copper will be plated upon the cylinder in a thin coherent coating which, if current of electricity has been properly regulated and amount of copper in solution not too great, may be washed without the slightest danger of loss. The solution must not more than two-thirds fill beaker nor must the cylinder be completely covered. When solution becomes perfectly colorless, a little water is added to beaker to raise the level of the liquid and so expose a fresh surface of platinum to its action. If no further deposit is made on the cylinder the precipitation is complete, but if a film of copper forms upon it the current must be continued until no more copper remains in solution. The copper is all precipitated if a few drops of the liquid on a watch-glass give no coloration when touched with a drop of sulphureted hydrogen water. Now slightly acidulate some water with sulphuric acid and, without interrupting current, displace liquid in beaker with the acidulated water by pouring it into a funnel which touches the bottom of beaker, again displacing the acidulated liquid with pure water; a porcelain dish can be placed under beaker to catch the liquid which overflows.

The cylinder and spiral are now disconnected from battery and the cylinder put into a porcelain capsule and washed by pouring hot water over it twice and alcohol once. The capsule is then heated a few minutes over the water-bath and when dry, cooled, and the copper plated cylinder weighed on an assay balance. The copper is dissolved off with nitric acid, and the cylinder washed, dried, and weighed. The difference between the two weights is the weight of copper in amount of sample taken. If several cylinders are used it will save time to number them and record their weights, as then it will not be necessary to weigh them every time.

Sometimes new foil will not act well; in that case scour it well with sea sand to remove the outer glaze, and if that is not effectual, a few minutes treatment with nitrohydrochloric acid will remove all difficulty.

If, on account of too much nitric acid having been used, the copper fails to precipitate, the acid may be partially neutralized with carbonate of soda and the solution somewhat diluted. If too dilute, or too little nitric acid is present, the precipitate will be spongy, which may cause a loss of copper. Any deposit on the spiral (lead, manganese, etc.) may be disregarded. Metals interfering by being deposited with the copper, are silver, antimony, arsenic, mercury, and bismuth. Silver may be removed by the addition of a very little common salt to the nitric acid solution before evaporating. Every trace of chlorine must be afterwards driven out with nitric acid, or the platinum cylinder might be attacked and results so vitiated. If the solution has been made in nitric acid only, not more than a trace of antimony can be present. Arsenic is not

frequently deposited on the copper, but even when it is, all the copper generally comes down first, when the current can be interrupted and the arsenic left in solution. It may be advisable sometimes to roast ore in order to volatilize arsenic, as well as quicksilver. Quicksilver, if present, would be deposited as a white coating upon the copper; in fact, from some experiments I have made, I think that an accurate mercury determination can be based on this method. Bismuth is not of frequent occurrence, but if present must be removed. It may be precipitated from the solution with carbonate of ammonia, the filtrate evaporated to dryness, ignited to remove ammonium salts, and redissolved with nitric acid, etc., as above. With size of foil given, the solution should not contain more than 0.4 gramme copper; so, in operating upon metallic copper, borings, etc., of which the direct solution of a small amount might not give a correct average, the portion used for assay should be obtained by dissolving from 2 to 10 grammes of the sample, filtering into a measuring flask, diluting to mark, and removing with a pipette from the uniform solution, a volume containing not more than 0.4 gramme pure copper.

TIN ORES.

TIN stone (tin oxide) is readily smelted to a button.
Take—

Powdered ore.....	1 part
Potassium Cyanide...	$\frac{1}{2}$ "
Soda.....	$\frac{1}{2}$ "

Mix; put in pot; heat quickly. When fused, leave five minutes in fire; then shake, tap, allow to cool, and break the pot. The button is silver white, malleable. A piece of the metal boiled in nitric acid forms a white powder insoluble in water. Antimony, which can be smelted in the same way, acts similarly in nitric acid. But antimony is neither white nor malleable. It is gray, crystalline, and brittle. (See Part I for distinction.)

The presence of quartz (silica) causes a large loss of tin in the smelting, hence, if the ore is not pure, it should be washed by "panning," or vanning, in order to remove the quartz as far as possible from the heavier tin oxide before smelting.

Mixed ores containing tin, or very impure tin ores, should be boiled, in the state of fine powder, in a mixture of three parts hydrochloric and one part nitric acid.*

*From Mitchell.

Heat gently for half an hour, then boil until the greater part of the acid has evaporated. Cool, add water, settle, and pour the water off, not losing any solid matter. Wash thus until the water is tasteless; the residue is tin oxide, quartz and tungstic acid if the ore contained tungsten, as it often does. The tungstic acid is removed by gentle heating for an hour with ammonia, stirring occasionally. The ammonia is then poured off and the residue washed. The quartz is removed by vanning, and the tin oxide smelted as before.

MERCURY ORES.

THE principal ore of mercury is the red cinnabar for which red iron-oxide is so often mistaken. There are also black ores of mercury, and many silver, copper, and other ores contain small quantities of mercury.

Before assaying a doubtful ore for mercury it is well to test it. Boil some of the powder in a mixture of nitric and hydrochloric acid; dilute and place a drop of the liquid on a piece of gold. Touch the gold, through the drop, by a pointed iron or steel implement, such as a penknife or a needle. If the ore contained a trace of mercury, the gold will be instantly amalgamated on that spot, which will be whitened. Or take a glass tube about six inches long and a quarter inch diameter, closed at one end; place in the tube about a grain of the ore mixed with two or three grains of litharge. In order to get the charge to the bottom or closed end of the tube without soiling the other part, line the tube with a rolled slip of writing paper (or glazed paper), introduce the charge and tap it down, then remove the paper. Heat the end of the tube containing the charge in the flame of a spirit lamp, or a candle. If mercury is present, it will first evaporate, and then condense in the cool part of the tube, in the form of minute globules which appear like a mist if the quan-

tity is very small. Cut the tube a little by a file near the charge, and break off the part containing the mercury. By means of a wire and a bit of moist cotton cloth, wipe the interior of the tube. The globules of mercury will be visible on the swab, by the aid of a magnifying glass, or may be washed off in a cup of water. By weighing the ore, using a longer tube kept cool by a moist cloth toward the open end, and carefully collecting and weighing the mercury, the operation becomes an assay.

The assay—

Ore.....1 part

Lime.....1-3 “

Charcoal.....1-10 “

Mix; put into an iron or clay retort; cover with lime and charcoal; close the retort and heat to bright redness, keeping the retort pipe cooled by a stream of water flowing onto a cloth wrapped around it. Receive the condensed mercury in a vessel of water placed under the end of the pipe. The pipe should not dip into the water, but a piece of cloth tied around it may. Iron filings or borings will answer in place of lime. Use one part to two of ore for the mixture, and some more as a cover. Any mercury which may remain in the pipe must be washed out. Pour the water off from the mercury; dry that by means of filter paper, weigh, and report in percentage of the weight of ore taken.

A small assay may be made in a tobacco pipe used as a retort, being closed by means of clay. A small amalgam retort is suitable. A quicksilver flask with a pipe adapted will answer for several pounds of ore. The flask should be previously heated to redness to drive out any quick-

silver which may be adhering to it, owing to its having contained quicksilver before. Gaspipe alone may be employed. A short piece, plugged at one end, serves to receive the charge, and this is connected by a sleeve with a longer piece. It is better if the short piece is of the greater diameter, say two inches, and connected by a reducer with a longer piece of $\frac{3}{4}$ -inch pipe. When the distillation is finished, the pieces are disconnected and the longer one washed for recovery of any adhering mercury.

ZINC ORES.

GENERAL METHOD FOR ALL ORES.—Treat from 10 to 100 grains, or a suitable number of grammes of the finely powdered ore with equal parts of nitric, hydrochloric, and sulphuric acid, added one after the other in the order named, in a porcelain vessel covered as usual, or in a matrass. Boil until heavy white fumes come off; cool, dilute, cover and pass in a stream of hydrogen sulphide until the liquid retains the smell. If arsenic is present, the solution must be boiled for some time with addition of sodium sulphite and kept acid by adding hydrochloric acid; otherwise, zinc will go down with the arsenic. Set it in a warm place until settled. Filter, and wash, with water containing hydrogen sulphide, as quickly as possible. Boil the liquid until it no longer gives off an odor of hydrogen sulphide; add a little nitric acid and again boil. To the hot liquid add a few more drops of nitric acid; if a dark shade is produced, more nitric acid is required. Boil down considerably, and if any sulphur is seen, oxidize it by a few crystals of potassium chlorate, or by boiling down with nitric acid. Cool; add ammonia in considerable excess. Filter, and wash with water containing ammonia as long as the drippings give a p. p. with ammonium sulphide. To the filtrate add

hydrochloric acid until it reddens litmus paper, and then sodium acetate in liberal quantity.

Again pass hydrogen sulphide, which will now precipitate the zinc as a white sulphide. Settle; collect the p. p. on a filter and wash with water containing hydrogen sulphide. Dissolve the p. p. by pouring hot dilute hydrochloric acid upon it, a little at a time, keeping it covered by a plate of glass, or better by a small funnel inverted. Wash the cover and filter with hot water. All the liquid is to be received in a clean beaker.

The treatment now depends on whether the last p. p. was purely white or mixed with black sulphide arising from the presence of nickel or cobalt in the ore,* or, more likely, from imperfect separation of copper at an earlier stage. In the first case, heat the liquid and add solution of sodium carbonate as long as it produces a p. p. Collect the p. p. which is zinc hydrocarbonate, on a filter, and wash it with hot water as long as the drippings cause turbidity in a little neutral solution of copper sulphate (bluestone), in a test-tube. Dry the filter with its contents; transfer the latter to a porcelain dish, and calcine thoroughly at a red heat, which converts it to zinc oxide. Weigh, and calculate 80.2 per cent. as zinc. In the second case, add solution of caustic potassa or soda in excess, which will redissolve the zinc oxide at first precipitated, leaving the other metals insoluble. Filter, and wash till the drippings no longer blue litmus paper. Acidulate the filtrate with hydrochloric acid, add sodium carbonate, and finish as in the first case.

* The precipitation of nickel and cobalt may be prevented by addition of citric acid.

A chemist can modify and simplify this process in the absence of certain metals, such as copper, arsenic, manganese, but these metals are frequent companions of zinc in our silver and lead ores, and it is solely on account of the influence of zinc on the selling price of these ores that I have thought it best to treat of the assay of that metal, which has no market value in such ores. As it would occupy too much space to give all the modifications applicable to different zinc ores, that process is given which is believed to cover all cases.

The trouble of igniting the p. p. may be avoided. Proceed as directed up to the point at which the precipitated zinc sulphide has been dissolved in hydrochloric acid. To the solution add ammonia in excess, then hydrochloric acid until it *very slightly* reddens litmus paper. Now add solution of sodium phosphate as long as a p. p. is produced. Heat nearly to boiling until the p. p. settles. Filter; wash with hot water until the drippings no longer cause turbidity in solution of silver nitrate. Dry at heat of boiling water (on the water-bath); remove from filter; weigh. It comes off the filter beautifully. Calculate 36.39 per cent. of the weight as zinc.

NEW METHOD.—This has given very fair results. Dissolve a weighed quantity of the ore as directed for the new copper assay, removing arsenic by magnesium mixture if but little iron is present, using ammonia, regardless of the presence of manganese. To the filtered ammoniacal solution add potassium zanthate as long as a p. p. is formed. Filter and wash. To the liquid add ammonium sulphide as long as a p. p. is produced. (The p. p. should be white, if not, proceed as in similar case where

hydrogen sulphide is used.) Filter, and wash with water containing a little ammonium sulphide, and lastly with hot water. Dissolve the zinc sulphide in warm dilute hydrochloric acid, saturate with ammonia, again add hydrochloric acid to *faint* acid reaction, then sodium phosphate, etc., as before.

NEW ASSAY OF NICKEL AND COBALT.

NICKEL and cobalt usually occur together. The substance may contain, besides these metals, iron, manganese, zinc, copper, gold, silver, lead, bismuth, antimony, tin, arsenic, tellurium, calcium, barium, strontium, aluminum, magnesium, silica and sulphur. Should it also contain mercury, that is easily expelled by calcination of the weighed assay before proceeding further.

Powder very finely, unless the substance is an alloy, when it may be cut or rasped. Take ten grains or one gramme, or less if the substance be rich. Digest in a plenty of nitrohydrochloric acid, the nitric in excess. Let all sulphur be dissolved, or remove the drops from the sufficiently cooled liquid, burn them in a small basin, dissolve the residue, and add it to the main solution. Add a little sulphuric acid, unless the assay contained a good deal of sulphur, as copper or iron pyrites. Evaporate nearly to dryness; cool; dilute; filter; wash with hot water until the drippings no longer give a dark p. p. with ammonium sulphide. (Test a small drop on white porcelain.) If the volume of liquid is now inconveniently large, reduce it by evaporation. Add solution of caustic potassa or soda very carefully, not producing any distinct p. p., although in many cases a slight turbidity, but the liquid must remain acid to test paper.

If too much alkali be added, it must be counteracted by a drop or two of hydrochloric acid; it is better to use no alkali than too much.

Now add moist precipitated zinc carbonate; stir, and continue adding the carbonate until a considerable excess of it remains unchanged in the liquid, which may be slightly warmed. If the assay contained no copper, test a minute drop on white porcelain, with a drop of solution of potassium sulphocyanide; when this gives no red or pink tinge, the iron is all down. If copper is present, test for that, in the same way, with potassium ferrocyanide, which will give a brownish red color as long as any copper remains in solution. As the iron goes down first, it is not necessary to test for that when copper is present; when the copper is all, or nearly all down, a bluish or greenish p. p. may be produced by this test, which must not be mistaken for the blue which a persalt of iron would produce; it is caused by cobalt or manganese.

When the copper is all down, filter, and wash with hot water as long as the drips give a p. p. with solution of potassium zanthate. (Return these tests to the filtrate.) For a final test of the washings, place a drop on porcelain and add a drop of ammonium sulphide; it should not blacken. Now warm the liquid to about blood heat, or a little higher, and add solution of potassium zanthate; stir, and continue adding the zanthate, at intervals, as long as it is seen to produce a yellow or a greenish p. p. Keep the liquid warm, and let it stand some time, stirring occasionally, until it clears tolerably. Try a little more zanthate; it will probably make a white p. p. of zinc zanthate which, however, either changes its color or disap-

pears, being either converted to nickel or cobalt zanthate, or dissolved. At last, filter a little of the solution, on a filter which has been exactly balanced with another on a fine balance. Test the filtered liquid with potassium zanthate; if any yellow or green p. p. forms, return the test to the main solution, and add more zanthate to that.

When no more p. p. forms, or only a white one which dissolves, make a final test of a drop with ammonium sulphide; it should give no black p. p. although a white one of zinc, perhaps tinged by pinkish manganese sulphide. Collect the p. p. after letting it settle, on the same filter that was used in testing. Any p. p. which may adhere to the side of the beaker must be removed by the aid of hot water and a rubber tipped glass rod. A short piece of pure rubber tube on the end of the rod will answer. Wash several times with hot water.

Place a clean beaker under the filter, and leach the p. p. with slightly diluted ammonia until that passes colorless, and a drop of it gives no p. p. with a drop of acetic acid (or *very* dilute sulphuric) enough to destroy the smell of the ammonia. (Return the drop, if it gives a p. p.)

Remove the beaker containing the ammoniacal solution of nickel zanthate, and wash the cobalt zanthate on the filter with hot water containing a little ammonia, until the filter is clean and the p. p. separates well from it under the jet. Dry the cobalt zanthate, still on the filter, and the counterpoise filter with it, on the water-bath; cool and weigh it, on the filter, putting the counterpoise filter on the weight pan; return both to the water-bath for ten minutes; cool and weigh again, and so until it weighs twice alike.

The cobalt zanthate should be of a dark, pure, but not brilliant green color. If mixed with mustard yellow, it contains a considerable proportion of copper. When calcined, it should be black, not green, which indicates the presence of zinc; and if it gives a blue solution with ammonia, copper is present. The copper may be removed thus: Redissolve the calcined p. p. and the burned filter, in aqua regia; dry with addition of hydrochloric or sulphuric acid; take up with water and a little of the same acid; digest warm with a piece of zinc, keeping the liquid distinctly acid so that bubbles rise from the zinc. The copper will be precipitated. Remove the zinc and copper, nearly neutralize the liquid by potassa, reprecipitate the cobalt by potassium zanthate, wash, dry and weigh.

To the ammoniacal solution of nickel zanthate, add *very* dilute sulphuric acid until the greater part of the nickel zanthate is reprecipitated, but the liquid still smells of ammonia; then add acetic acid until the liquid has a slight acid reaction. Stir, to curdle the p. p.; let stand for some time, stirring occasionally. Add a little potassium zanthate, and if it produces a p. p. add as much more as may be necessary, but avoid great excess. Filter, cleaning the beaker as with the cobalt; wash with hot water until the p. p. cleaves readily from the filter under the jet. Dry on the water-bath; transfer from the filter to the weighing capsule; weigh; place the capsule with the p. p. on the water-bath for 15 minutes; cool, weigh, and so on until it weighs twice alike. A tared or counterpoised filter may be used, as with the cobalt, but the nickel zanthate generally comes off the filter cleanly if well washed.

Each of the zanthates contains 19.6 per cent. of metal

or, the weight multiplied by .196 is the weight of the nickel or cobalt. The filtrations may be greatly facilitated by connecting a foot of rubber tubing to the beak of the funnel and letting it hang downward, the end in the receiving vessel. This should not be done while leaching with ammonia.

In certain circumstances, believed to be the presence of arsenic in the solution, the precipitation of copper by zinc carbonate is not quite complete. If, however, there is sufficient iron present, the difficulty no longer exists; hence, if a little copper should remain in solution, notwithstanding an excess of zinc carbonate, add a little iron perchloride, as nearly neutral as may be, and then more zinc carbonate if necessary. If the ore is known to contain arsenic, with but little iron, while copper is also present, it may save trouble to add a little metallic iron to the acid solution, before treating it with zinc carbonate, and digest warm until the iron is dissolved. In this way the quantity of copper in the solution is reduced and that of iron augmented. The liquid must be kept distinctly acid during this operation, so that bubbles of gas rise constantly from the iron. The iron must be peroxidized. In the treatment with zinc carbonate, the liquid may be slightly warmed, not highly heated.

The potassium zanthate made, as directed for the copper assay, with alcohol of 95 per cent. or thereabout, is a little alkaline, and becomes more so by keeping. In this state it is not fit for this assay. Before using it, test it with faintly reddened litmus paper; if it blues that, add some very dilute sulphuric acid, not strong enough to cause turbidity, or, if it does so, warm the liquid until clear. Or, add

carbon bisulphide and alcohol to dissolve it until the liquid is neutral.

To make the zinc carbonate, or rather hydrocarbonate, dissolve zinc sulphate in water, and filter it. Also dissolve sodium or potassium carbonate in water. Filter the latter into the former until nearly but not quite all the zinc is thrown down. Collect the p. p. on a filter and wash it. The filtrate should still give a slight p. p. on addition of the soda solution in order to guard against an excess of soda, which would be difficult to wash out of the zinc carbonate. Keep the carbonate moist in a wide-mouthed jar or bottle.

Resume of Operations.

1. Dissolve; dry; take up; filter.*
2. Treat with zinc carbonate; filter.
3. Precipitate with potassium zanthate; filter.
4. Leach with ammonia.
5. Dry and weigh cobalt zanthate.
6. Recover nickel zanthate by acid; filter, wash, dry, weigh.

7. Multiply weights by .196 for metal. If the greatest possible accuracy is required, see *Fresenius' Quan An*, or send the assay to an analytical chemist.

"While working up this assay, I was informed that a Mr. Phipson, in 1877, had published an account of the precipitation of nickel and cobalt by potassium zanthate, and the separation of the zanthates by ammonia. So far as I know, however, Mr. Phipson did not determine the metals quantitatively as zanthates, nor apply his method to the assay of ores.

* This filtration may probably be omitted without injury.

"As I wish to be perfectly fair and honest in these matters, I will state that my knowledge of the different behavior of nickel and cobalt zanthates toward ammonia was the result of experiments made by Mr. G. F. Beardsley, in consequence of some information I had given him on copper and nickel zanthates.

"Conceiving the idea that the reactions might be made use of in the assay of the three metals, I took the first opportunity, which did not occur until some years later, of working them up, with the results given in this book. My main object was to avoid the use of hydrogen and ammonium sulphides.

"I am not aware that the copper assay by zanthate has ever been attempted before, and, taking my assay of nickel and cobalt as a whole, it is certainly original, and I think myself justified in calling it new."

ASSAY OF CHROMIUM.

THIS assay is not for every substance which may contain chromium, but for chrome ore proper. Powder as finely as possible. Take 5 grains or half a gramme. Fuse in a platinum crucible with 2 parts of nitre and 4 of soda, or better with dried caustic soda alone. Add the soda, etc., a little at a time, and stir with a stout platinum wire. Cool, and dissolve in hot water (put crucible and all into the dish), filter; acidulate with hydrochloric acid; add alcohol and boil until the liquid becomes quite green. Precipitate by ammonia, and boil. Filter while hot, and wash with hot water. Dry the p. p.; burn the filter, and add the ashes to the p. p. Ignite the whole. Weigh; deduct the weight of the ashes of a similar filter. Report as so much per cent. chromium sesquioxide, which is what the p. p. consists of. The stirring with platinum wire during the fusion often prevents the need of a second fusion, but if any ore remains insoluble, that portion must be collected, dried, and fused with a fresh quantity of soda, etc.

The platinum crucible must not come in contact with the fuel, therefore put it inside of a common assay crucible and pack lime around it.

ASSAY OF BISMUTH.

SMELT a suitable quantity of the powdered ore in an assay crucible, with borax and a mixture of equal parts of soda and potassium cyanide. Copper may be kept out of the button almost entirely by means of sulphur or arsenic; hence, if the ore contains copper, and little arsenic or sulphur, or none, add sulphur and powdered charcoal in the dressing, also a couple of nails. In this way the bismuth can be got tolerably pure from very complex ores containing copper, zinc, nickel, cobalt, and tellurium. Gold and silver, if present, will go into the button, which should therefore be cupelled after weighing, and the weight of precious metal found deducted.

Lead and antimony, if present, will go into the button. With very poor ores, it is even advisable to add some litharge in the dressing, in order that the resulting lead may collect the bismuth. About 4 times the weight of the bismuth will suffice. In these cases, or if much copper has been allowed to go into the button, that must be purified. Laminate or powder it, according as it is tough or brittle, and digest it in boiling nitric acid, which must be considerably diluted if lead is present. This will dissolve the bismuth, lead, copper, and silver, leaving antimony (and tin) insoluble. Add enough sulphuric

acid to precipitate the lead; boil off some of the acid; cool; add water and boil. If there is any insoluble matter, settle; decant onto a filter; boil again two or three times with water and a little sulphuric acid, also a drop of nitric acid, decanting each time onto the filter. Finally throw the residue onto the filter and wash it slightly; it should be tested by blowpipe for bismuth which should not be found.

To the filtrate, in a large, covered beaker, add excess of ammonium carbonate; heat to boiling; filter; wash with water containing ammonia until that passes colorless. Dry; remove the p. p. from the filter, which burn. Smelt the p. p. and the filter ash together, with a little borax glass and potassium cyanide, in a small crucible. Cool and break the pot; separate the button from the slag with care; it may be cleaned by means of warm water with a little sulphuric or hydrochloric acid.

ASSAY OF ARSENIC.

IN a porcelain crucible fuse half a gramme (or less if rich) of the powder with 6 to 10 parts of a mixture of sodium carbonate and nitre (in equal parts). Cool; extract with water warmed in the crucible; filter. The filtrate contains the arsenic combined with alkali. Acidify with nitric acid, and boil. Cool; neutralize almost exactly by ammonia, so that previously reddened litmus shows alkali in half a minute. If not clear, filter. Add solution of neutral silver nitrate (lunar caustic) in slight excess. Stir to coagulate the p. p., which is of a brick red color. Filter; wash the p. p. with cold water. Test the filtrate with silver nitrate, dilute nitric acid and ammonia (to again neutralize), in order to see that precipitation is complete. Scorify the p. p. with lead, and cupel the button.

If the ore contained any chlorides, as silver chloride, etc., instead of at once scorifying the p. p. pour dilute nitric acid on it, on the filter, to dissolve it and separate it from silver chloride which will remain on the filter. To the liquid add enough hydrochloric acid to precipitate all the silver in that as chloride, which collect on a filter, wash with hot water, dry and scorify with lead, adding a little soda. Calculate the arsenic from the silver got, in the ratio of 648 of silver to 150 of arsenic.

The writer has not tried this method, but it seems to be a good one. It is stated that phosphorous and molybdenum may interfere.

ASSAY OF ANTIMONY.

FUSE a weighed quantity of the powdered ore, in a clay crucible, with 4 parts potassium cyanide, at as low heat as possible. Cool and break the pot. Carefully separate the slag from the button, which will be brittle almost invariably. If the ore contained little or no other reducible metal, the button may be weighed at once as metallic antimony; otherwise, as in the presence of lead, etc., powder it if brittle, laminate if tough, and digest with rather dilute nitric acid. Nearly all of the antimony will remain as a white powder which, when washed with hot water, dried, and again fused with potassium cyanide, will yield a button of metallic antimony (and tin, if present, which is not probable).

To assay an alloy of antimony and tin, dissolve in aqua regia with excess of hydrochloric acid, dilute the solution, adding more hydrochloric acid if a white p. p. falls, to redissolve that. Place a piece of pure tin in the liquid, keep warm, and maintain a slight excess of hydrochloric acid. Collect, wash, dry and weigh the precipitated antimony. The lost weight will be that of the tin in the alloy. These methods are not quite exact but will generally serve the purpose of the assayer.

ASSAY OF SULPHUR.

ACT on a weighed quantity of the powdered ore with aqua regia, or strong nitric acid and potassium chlorate. If some sulphur separates in clear melted drops, cool sufficiently, remove the solidified drops and weigh them. Evaporate to dryness, adding some hydrochloric acid toward the end. Boil with strong solution of potassium carbonate, or, but not so well sodium carbonate, for half an hour. Filter and wash; acidulate the filtrate, in a large beaker, with hydrochloric acid, add solution of barium chloride in slight excess. Allow to settle in a warm place for an hour or more, filter on a fine filter, wash with hot water, dry, and ignite the p. p. Weigh when cold, and multiply the weight by 0.137 for weight of sulphur. Add the weight of any sulphur that may have been weighed as drops. If barium is present, fuse the residue in a platinum crucible with 5 parts of mixed sodium and potassium carbonates; cool; dissolve in water, filter and wash. Acidulate the liquid and add it to the other.

ASSAY OF SALT.

IN buying salt for milling purposes it is often désirable to know its percentage of purity, or its fineness.

Take 542 parts by weight (say 542 grain or gramme points = 5.42 grains or 0.542 gramme). If it were pure, it could convert $1,000 = 10$ grains or 1 gramme of silver into chloride. Dissolve in water; acidulate slightly with nitric acid; warm and add solution of silver nitrate; stir to curdling of the p. p.; settle and add a little more silver nitrate, and so as long as a p. p. is formed. An excess of silver nitrate will do no harm. Settle; filter; wash with hot water until the drippings cause no p. p. in salt solution. Dry; dress on filter with litharge, soda, and borax mixed. Put filter and all into a crucible; smelt and cupel as in an ore assay. Weigh the resulting silver bead, adding a correction for cupellation loss according to experience. The bead weight in points is the fineness of the sample, and that divided by 10 is the percentage of pure salt (or the equivalent of that for utility).

Marsh salt often contains soda or lime, and, if used in conjunction with bluestone or acid, as in working silver ores by amalgamation, renders a portion of these useless. In acidulating the solution as above, observe if much effervescence ensues, as it may be better to pay a higher price for a better article.

To find how much bluestone the impure salt will counteract, take 100 parts (say grains) of the sample; dissolve in water and add 10 parts of bluestone also dissolved in water. Boil for a few minutes, and if the liquid is decolorized, add another quantity of bluestone and again boil. Filter and wash; acidulate the liquid with sulphuric or hydrochloric acid; precipitate, wash and weigh the copper remaining in solution exactly as in making a copper assay (or determine the copper by either of the other processes given). The weight of the copper multiplied by 4.06 is the weight of bluestone not made useless by the impurities in the sample, and this deducted from the total weight of bluestone used leaves the weight made useless by 100 of the sample. The blue crystals of bluestone should be used, rejecting any white portion.

To find out how much of the acid in use the material will neutralize, take 100 or 1,000 grains of the sample; dissolve in water and heat the solution. Take also a weighed quantity of the acid, say 10 or 100 grains, and pour it into 5 or 6 times as much water;* mix and weigh again, or measure the mixture, if more convenient. Add the acid liquid, little by little, to the salt solution, stirring, and, when effervescence ceases, testing after each addition with blued litmus paper, until that is slightly reddened. The proportion of the dilute acid used indicates the proportion of the acid taken which was necessary to counteract the alkaline matter in the taken weight of the sample.

*Never pour water into strong acid, but pour the acid into the water to avoid explosion.

APPENDIX TO PART I.

A NEAT way in which to make the assay of gold and silver ores containing much copper, is to treat the weighed assay with strong nitric acid until the copper is dissolved; filter and wash. To the filtrate and washings add hydrochloric acid or solution of common salt as long as a precipitate is produced. Collect the p. p. on a separate filter and wash it. The whole of the silver will be on the filters, that portion which existed in the ore as chloride, iodide or bromide on the first, the remainder on the second, also converted into chloride. It will not answer to add salt or hydrochloric acid during the treatment of the ore, because the proper quantity cannot be known, and an excess would cause gold to be dissolved and lost.

Dry the two filters with their contents; remove them from the funnels; dress the residue from the ore with litharge, borax and soda as a crucible assay by the first system, proportioning the fluxes to the quantity of substance. If there should be much separated sulphur, some nitre may be needed. Put the whole into a crucible. Dress the other filter with a small quantity of litharge and soda, and put it also in the pot. In adjusting the flour or nitre, it must be remembered that the filters will act as reducers, unless indeed they have been burned to

ashes in the drying, which may be done on a roasting dish in the muffle very speedily. Cover with salt, and on that put some borax. Melt and proceed as usual. Assays made in this way work very cleanly and quickly, as there is not much besides quartz and silver chloride to melt. The method has not, however, usually given any better results than others. The chief advantage is that a very large quantity can be taken, it being greatly reduced by the acid treatment. In the case of sulphuretted ores, time may be saved by roasting the weighed assay before dissolving the copper out.

In assaying coppery ores by the second system, it should be remembered that soda, in presence of carbon, can reduce some metallic copper from the sulphide, for which reason flour or charcoal should be used only in such quantity as to deoxidize the litharge, copper oxide, etc. It may be omitted entirely if enough sulphur is present or added.

For concentrated pyrites, whether concentrated naturally or artificially, the second system is decidedly preferable in point of convenience. In assaying this class of material by the first system, using nitre to counteract the excess of reducing matter, it may happen that, although the nitre has been adjusted in accordance with the indications of a preliminary, little or no lead is got. The natural supposition then is, that too much nitre has been used, and that the lead remains in the state of oxide. This is an error. The lead is retained not by oxygen but by sulphur. On repeating the assay with a smaller proportion of nitre, the result is no better than before, while, if it be made with a larger proportion, the apparently paradoxical result is that more lead is got, though not the full quantity called for originally.

This thing has puzzled many an assayer and is understood by very few. It has led some to the practice of using nails in a nitred assay at the risk of getting too much lead, and of forming oxysulphides in the slag, which is stated on good authority to be dangerous. It is true that I have suggested the use of a *small* quantity of nitre in the assay of such ore by the second system, but the litharge used in that is not in excess of the quantity of lead required, and too much cannot be got, while the little nitre intended to be used, if any, is only enough to burn a portion of the *excess* of sulphur which exists in iron pyrites beyond what is required to retain the iron in the form of a sulphide containing the minimum proportion of sulphur, to which condition it must be reduced in order to ensure the extraction of the precious metal. It is for this purpose, in part, that nails are used.

The difficulty spoken of is caused, not by an excess of nitre, but by a deficiency of litharge. It has already been stated that iron pyrites require 50 parts of litharge to effect complete oxidation, reducing only 8 or $8\frac{1}{2}$ parts of lead, that is very much less than the entire quantity of lead in the litharge. The reason of this is, that when litharge is combined with a certain proportion of iron oxide, it loses the power of oxidizing sulphurets. When the ore consists entirely of iron pyrites which, in this class of assay, must be completely oxidized, a large quantity of iron oxide is formed. This engages a corresponding quantity of the litharge, rendering that powerless to decompose sulphurets of any kind, even galena. Now suppose we use nitre enough to oxidize about 90 per cent. of the pyrites, the remaining 10 per cent. (the ore being all pyrites) re-

quire 50 times as much litharge, that is 5 times the weight of the entire assay. Besides this, a quantity of litharge is required to combine with the iron oxide produced by the action of the nitre on 90 per cent. of the pyrites, otherwise the 10 per cent of pyrites cannot be oxidized, because the litharge intended to act on them is otherwise employed. The result is that little or no lead is got, since little or no litharge is reduced. It remains in the slag partly combined with iron oxide and partly with a portion of the sulphur of the 10 per cent. of the pyrites.

It can now be seen why more nitre may cause more lead to be reduced by leaving less than 10 per cent. of the pyrites to be acted on by the litharge, for, although more iron oxide is produced, it does not consume litharge in the same proportion as is required by the pyrites. The remaining quantity of the latter is then completely oxidized, and an equivalent quantity of lead is thrown down. If a sufficient quantity of litharge had been used, instead of more nitre, the 10 per cent. or so of pyrites would have been oxidized, and the calculated quantity of lead got. The indications of a well-made preliminary may always be relied on if enough litharge is used.

From the foregoing it appears that the assay of this class of ore by the first system, using a graduated quantity of nitre, must require even more than the 5 parts of litharge prescribed for all ores by Mitchell, and this is true when the ore is all pyrites. But it seldom happens that even concentrations contain more than 80 per cent. of pyrites, the remainder consisting of other sulphurets, quartz, etc. Moreover, as has been stated heretofore, the presence of soda and silica, forming glass, and of borax

also, has an influence because these combine with the iron oxide, releasing a corresponding quantity of the litharge, so that the more of them we employ, the less litharge is necessary.

For well concentrated pyrites, the following dressing is suitable.

Ore.....	1	part
Nitre.....	1.7	"
Litharge.....	8-10	"
Soda.....	1-2	"
Borax.....	1	"
Glass.....	$\frac{1}{2}$ -1	"

The litharge may be lessened as the proportion of soda and glass is increased.

The following described experiments which I made for the purpose, will assist in understanding the facts stated.

1st.	Ore.....	12 grains
	Litharge.....	480 "

Gave, on fusion, 63 grains of lead. A larger proportion of litharge gave no more, the ore not being pure pyrites.

2d.	Ore.....	12 grains
	Litharge.....	240 "
	Soda.....	240 "

Gave also 63 grains of lead.

3d.	Ore.....	12 grains
	Litharge.....	240 "

Gave only 57 grains of lead which was not well collected in a button.

4th.	Ore.....	12 grains
	Litharge.....	240 “
	Glass.....	30 “

Gave 61 grains of lead, and poured cleanly.

5th.	Ore.....	12 grains
	Litharge.....	120 “
	Soda.....	240 “

Gave 47 grains of lead which was a good deal scattered.
Remelted with more soda, gave 48 grains lead.

6th.	Ore.....	12 grains
	Litharge.....	120 “
	Soda.....	120 “
	Glass.....	60 “

Gave 63 grains of lead and a clean pour.

7th.	Ore.....	120 grains
	Nitre.....	130 “
	Litharge.....	240 “
	Soda.....	240 “
	Borax.....	120 “
	Glass.....	120 “

Gave only 63 grains of lead, instead of about 120 for which it was nitred.

8th.	Ore.....	120 grains
	Nitre.....	130 “
	Litharge.....	480 “
	Soda.....	120 “
	Borax.....	120 “
	Glass.....	80 “

Gave only 60 grains of lead, but remelted with another ounce of litharge it yielded 64 grains additional. A ninth

trial, with similar proportions of nitre soda, borax, and glass, but with 2 ounces of litharge in the first melt, gave a satisfactory button which was not weighed.

In assaying arsenical ores by the second system, a large quantity of iron is consumed. The presence of soda diminishes the consumption of iron by sulphur, but not by arsenic. On breaking the slag, a double button is found, the one part hard and brittle, which is iron arsenide; the other soft and malleable, which is the lead. The two are easily separated. The arsenical button contains no precious metal, but if the ore contained nickel or cobalt, that will be found in the arsenide, which is one advantage of the second system over the first, in which nickel or cobalt may get into the button and give great trouble. Arsenic may be added for the express purpose of separating nickel and cobalt, just as sulphur is for copper.

When on pouring an assay, fluxed according to the first system, it is found that, although the slag is sufficiently liquid (or otherwise) the lead is scattered, beads of it clinging to the walls of the crucible, it indicates either that the assay has not been sufficiently heated, or that not enough litharge has been employed. The assay may be at once returned to the crucible from the mould, provided that no part of the slag has been lost by overflowing, and, remelted with the necessary correction, it can then be cleanly poured.

Some readers of Part I have objected to the instruction there given to heat slowly the assay by scorification; they prefer to put the assay at once into the highly heated muffle. Certainly, in the case of oxidized ores, slow heating would be a waste of time, and with some sul-

phuretted ores it may be unnecessary, but it is nevertheless a fact, as stated, that some ores cause sputtering if heated suddenly, and my aim throughout has been to keep on the safe side. An intelligent workman will soon learn to modify his work according to circumstances. Another suggestion is that a definite rule for the proportion of water to be used to moisten the bone ashes for cupels would be useful. Three fluid ounces to the pound of ashes will generally answer. Some mix sifted wood ashes with the bone ashes; others use a little soda.

WEIGHING BY THE OSCILLATIONS OF THE POINTER.

ON page 72, Part , will be found some instructions on this subject. There are, however, two methods in use, and this fact, together with the indefinite manner in which the terms designating the position and movements of the pointer are used by different writers and operators, has caused some confusion and misconception among students.

The one method is, to take the *excess* of movement on either side of zero as the indication of the excess of weight; the other is, to take the distance from zero at which the pointer will finally rest as such indication. The first method will give twice as many divisions of the arc as the second, hence the value of a division is but half as much in the first as in the second. The second is the method given in Part I.

In order to clear the subject of all ambiguity, I propose the following terms: Range, swing, and rest. Range is the total movement of the index, counted in divisions of the arc (incorrectly called *vernier* by some). Range may extend to each side of zero, and is then written + and -, plus being usually the right-hand side, minus the left, as $+5 - 3$. Or range may extend from any di-

vision on either side to zero, as $+5 - 0$, or $-6 + 0$. Again, the pointer may move to either side and not return to zero, as $+7 + 3$; that is, the pointer goes to $+7$ and comes back to $+3$, or $-7 - 3$ on the other side. Range has no value as an indication of the amount of preponderance, but it affords a means of calculating swing and rest.

Swing is the excess of movement to one side or the other of zero, caused by a preponderance of weight on one of the pans; hence a balance in equilibrium will have range, if the beam be set in motion, but can have no swing in the sense which I propose for that term. Swing is equal to range when one extreme of range is zero; it is the difference of the distances from zero when the pointer goes to each side of zero, that is when the signs of the distances are opposite; it is the sum of the distances when the signs are similar, because if the pointer marks $+5$ and comes back to $+3$, it would mark $+8-0$, if the beam had impetus enough, or $+9-1$, etc., in each instance giving swing = 8.

Range is a matter of observation only. Swing may be a matter of observation, for if impetus enough be given to the beam to make the pointer move to right and left of zero, a time must come when the gradual decrease of movement will, practically speaking, cause it to touch zero at one extreme, and when that is the case as said above, swing is equal to (identical with) range. But swing may be calculated from observed range at any time when the beam is oscillating, as shown above.

Rest is the distance from zero at which the pointer will stop if allowed to do so. In this sense, a balance in

equilibrium can have no rest, though, as before remarked, it may have range. Rest is always half of swing. It may be observed by waiting for it, or it may be known in advance by finding swing and dividing that by 2.

Swing, and therefore rest, may be deduced from a single observation of range, but more accurately from the mean of several, thus: note on a piece of paper three (or more) extremes in one direction, and the intervening two (or more) extremes in the other direction, prefixing to each its appropriate sign; find swing by adding the means together if the signs are similar, subtracting the lesser from the greater, if the signs are opposite. In the first case the sign of swing will be the same as that of range; in the second, it will be that of the greater quantity. Rest takes the same sign as swing.

Range may be increased whenever desired for convenience, by wafting with the hand a slight current of air against either of the pans, after which the case must be closed, and a few moments allowed to elapse before beginning the observation. Examples: Range (an average) is $+8-3$; as the signs are opposite, swing is the difference, $= +5$; rest is $+2\frac{1}{2}$. Again; range is $-8+3$; the signs are opposite and swing is -5 ; rest, $-2\frac{1}{2}$. Let range be $+7+2$; the signs are similar and swing is the sum $= +9$; rest is $+4\frac{1}{2}$. If range is $+6-0$, swing is $+6$, and rest is $+3$; or if range be $-6+0$, swing is -6 , rest -3 .

The value of swing or rest for the balance in use may be found by trial with a known preponderance of weight on either pan. It varies slightly with the load in the pans. Whether the preponderance of weight indicated by swing or rest must be added to or subtracted from the

weights in the weight-pan must be left to the common sense of the workman, since it depends upon which pan contains the object to be weighed. If, however, the signs are used as here suggested, and the object be on the left-hand pan, and the index of the balance points downward, the value of a + swing or rest must be added, that of — subtracted.

APPENDIX TO PART THREE.

THE ASSAY OF LEAD.

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IN the wet assay of lead, it is not strictly necessary to filter before the boiling with sodium carbonate. Digest with nitric acid, or with nitric and sulphuric; boil off a part of the acid; add sodium carbonate and boil twenty minutes; test the liquid with red litmus paper, which should show a strong alkaline reaction, otherwise more soda must be added and the boiling repeated. Filter and wash; dissolve the carbonate on the filter by pouring acetic or dilute nitric acid on it, receiving the solution in a clean vessel. To the liquid add dilute sulphuric acid so long as a p. p. is produced; warm, settle, filter, and wash with water containing sulphuric acid. Dry, ignite and weigh. If the ore is supposed to contain strontium, smelt the p. p. with potassium cyanide, and weigh the resulting button of metallic lead. In the fire assay, the stirring with an iron rod, as directed, is only necessary when the assay contains much arsenic, sulphur or antimony, the object being to avoid the inconvenience of the nails sinking beneath the slag. The heat should not be so high as in assaying for gold by the second system, but sufficiently so to make a liquid slag.

THE ASSAY OF COPPER.

IN assaying copper ores by precipitation on iron or zinc, it often happens that a sulphuretted ore as well as an oxidized one, if free from arsenic, will give clean copper without the treatment with ammonia. Nitric acid must be expelled by boiling down with hydrochloric or sulphuric acid, the latter if lead is present. When ammonia is used, the solution need not be first filtered unless much silver is present, in which case the assay should always be boiled down with sulphuric acid, because silver chloride is soluble in copper chloride. This applies to the new process as well as to others.

Sometimes the copper comes down black, while an examination of the ore by blowpipe fails to reveal any cause. Resolution and precipitation does not always give a properly colored precipitate. This may have been observed by other assayers, yet I find nothing about the cause or prevention in the books, that is granting that the solution is made as directed. I have succeeded in cleaning such black copper by re-dissolving in nitric acid, precipitating with excess of caustic potassa solution, boiling, filtering, again dissolving the black oxide in hydrochloric acid, and precipitating on iron. When the ore is known, by former experience, to be of this char-

acter, it will probably answer to treat the first solution with caustic potassa, filter, wash, and re-dissolve in hydrochloric acid, then precipitate.

The assay of copper by the new method may be made gravimetrically. Prepare the solution as before directed; precipitate the copper by a slight excess of potassium zanthate. If the p. p. should be brown, add more ammonia, and it will soon become yellow, unless cobalt is present, in which case proceed as before directed for such contingency. Settle, filter, wash, first with hot water, and then with alcohol of 95 per cent, until the drippings no longer become turbid on addition of water. Dry on the filter at the heat of boiling water. The p. p. may be removed from the filter, for the weighing, with but little loss, or it may be weighed on a tared filter. Calculate 33 per cent of the net weight, as copper, or, if not too particular, take one-third.

NOTE.—I regret that the system which I now propose, of weights to be taken for the assay of certain substances, did not occur to me until nearly the whole of the foregoing matter had not only been written, but also printed. There is an example of the method in "Leaching Gold and Silver Ores," in the "Assay of Manganese," and one in the "Assay of Salt," in the present book, and it was while thinking over the latter that the idea occurred to me of a general application. In all those methods, in which the substance got is different from that sought, the practice has been to state that the former contains so much per cent. of the latter, or the weight got must be multiplied by some number, which is often an inconvenient one, in order to ascertain how much of the substance sought was present in the assay.

It is much simpler to take such a weight of the assay substance as would, if it consisted wholly of the substance sought, give 100, by similar weight, of the substance to be got and weighed. The weight got would then be the percentage of the substance sought which the assay contained. In the wet assay of lead, for example, when not the lead itself but the sulphate is to be weighed, as lead sulphate contains 68.33 per cent. of lead it is easily seen that 68.33 parts of pure lead would give 100 parts of sulphate, and 68.33 parts of a substance which is only partly lead would give so much sulphate as would correspond to the percentage of lead in it.

In case the quantities should be inconveniently large or small, in the system of weights used, we may take a simple multiple or an aliquot part of the whole, and divide or multiply the weight got accordingly by some simple number.

Following are the weights of ore, etc., to be taken on this system to be assayed for the respective substances weighed in the forms named. The weights may be grains, hundredths of grains, grammes, milligrams, etc., as may be convenient; the weight got being stated in similar terms is the percentage of the substance sought.

68.3 for lead as sulphate.

36.5 for zinc as ammonio phosphate.

79.0 for copper as oxide.

33.0 for copper as zanthate.

19.6 for nickel and cobalt as zanthate.

13.8 for sulphur as barium sulphate.

23.15 for arsenic as metallic silver by substitution.

Whenever an assay of any kind is undertaken, a record, or at least a memorandum, should be made of the quantity operated on, and any other particulars which require to be kept in sight. In cases of rather complex operations it is a good plan to write down the entire proposed course of treatment, and to check off each step as it is performed; especially should this be done when variations from the prescribed course are contemplated, as will often occur if the workman is intelligent, and has some knowledge of chemistry.

When a multiple or aliquot part of a standard quantity is taken, the fact should appear on the record; for example, in the copper assay where the copper is to be weighed as zanthate, 33 grains would be too much of a rich ore, giving a too great volume of p. p. for rapid working. A tenth of the quantity will suffice in such a case, and may be written $\frac{33}{10}$ grains, in order to show that the weight of copper zanthate got in grains is to be multiplied by 10 for the percentage, because 33 grains of copper would give 100 grains of zanthate. On the other hand, if gramme weights were used, 33 grammes would be too much of any ore, and 33 milligrammes would be too little of any. In this case $\frac{33}{100}$ grammes = 330 milligrammes might answer, and should be entered in the form of a vulgar fraction as a reminder that the p. p. weight, in grammes, is to be multiplied by 100; or it might be 33×10 mgs., which would indicate that the p. p. weight, stated in mgs., must be divided by 10. A little practice will give facility in this system of weighing, and the rule for the weight to be taken is, that it should be numerically equal to the percentage of the

substance sought contained in, or indicated by, the substance got. In the assay of manganese for chlorination works, the substance sought is manganese binoxide; the substance got (weighed) is carbon dioxide, and 99 of the former are indicated by 100 of the latter, hence we take 99 of the assay substance. In the arsenic assay, as here given, arsenic is sought and silver is got; 100 of silver in this case indicate 23.15 of arsenic, and we take 23.15 or $\frac{23.15}{10}$ grains of the ore. In the copper assay, if we are going to precipitate the copper in the metallic state, copper is sought and copper is got, and copper of course contains 100 per cent. of copper, so we take 100 of ore in any convenient term of weights, or we take $\frac{100}{10}$ perhaps; but if we are going to get the copper in the form of oxide, containing 79 per cent. of copper, we take 79 of ore or $\frac{79}{10}$, 79×10 , etc., according to the estimated richness of the ore and the denomination of the weight.

The system is not applicable, or at least is not specially advantageous when two or more substances are to be determined from one assay quantity, unless all are to be weighed in forms which have equal percentage values. This is the case with nickel and cobalt when weighed as zanthates.

In conclusion it is proper to explain that methods are subject to modification, and even to total change, often with a saving of time and labor, under the varying conditions presented by difference of composition of the substance treated. I have given a few of these modifications; to give them all would be to write a good sized volume on each assay. My aim has been to give, first and more particularly, a process which is, as nearly as

may be, sufficient for any probable case within the scope of my plan; to enable the student to reach a reasonably accurate result, even though not in all cases by the shortest road. In the assay of lead or copper, for instance, there are many methods which could not be given in a book of the size and price of this. Those who desire to study other, and I freely admit, for certain cases, better processes, may find them described in other and more pretentious works. This book is not designed for the use of students who enjoy the advantages of professional instruction, but simply for that of people who want to assay, whether for business or pleasure.

Some defects in the arrangement of matter may be ascribed to the circumstance that considerable additions to the manuscript were made while the writer was at a distance of more than a thousand miles from the first written portions, and some, as well as alterations, while the work was in press.

My thanks are due the gentlemen of the Pacific Press Publishing Company for their efforts to oblige me under conditions which must have caused them some annoyance.

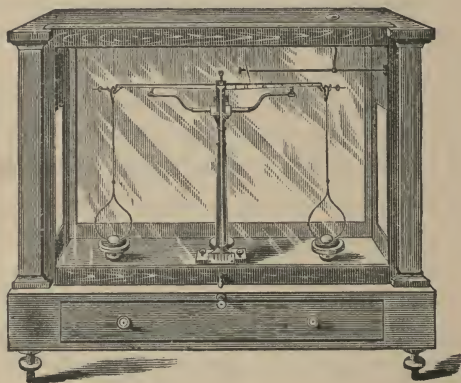
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A PLAIN BOOK ON ASSAYING.

"ASSAYING GOLD AND SILVER ORES."

BY C. H. AARON.

PART I.

The author of this work is well known in the mining regions of the Pacific Coast as a practical metallurgist of many years' experience. His writings for the press and his two previous works ("Testing and Working Silver Ores," and "Leaching Gold and Silver Ores") have shown his ability as a writer. The little book is plainly and simply written, more especially for the use of those persons not familiar with chemistry. No symbols are used, everything being plainly stated and clearly described. The scope of the book is shown in its table of contents, as follows: Introduction; Implements; Assay Balance; Materials; The Assay Office; Preparation of the Ore; Weighing the Charge; Mixing and Charging; Assay Litharge; Systems of the Crucible Assay; Preliminary Assay; Dressing the Crucible Assays; Examples of Dressing; The Melting in Crucibles; Scorification; Cupellation; Weighing the Bead; Parting; Calculating the Assay; Assay of Ore Containing Coarse Metal; Assay of Roasted Ore for Solubility; To Assay a Cupel; Assay by Amalgamation; To Find the Value of a Specimen; Test for Ores; A Few Special Minerals; Solubility of Metals; Substitutes and Expedients; Assay Tables. These assaying tables give simple directions for figuring out results. This is the simplest, cheapest and most easily comprehended work on Assaying yet published. The volume comprises 105 pages, with illustrations, and is well bound in cloth. The price is \$1, postpaid. Published by Dewey & Co., MINING AND SCIENTIFIC PRESS office, San Francisco—1884.

N. B.—The above refers to Part I, which preceded the issue of this book, *i. e.*, Parts II and III.

AARON'S LEACHING GOLD AND SILVER ORES.

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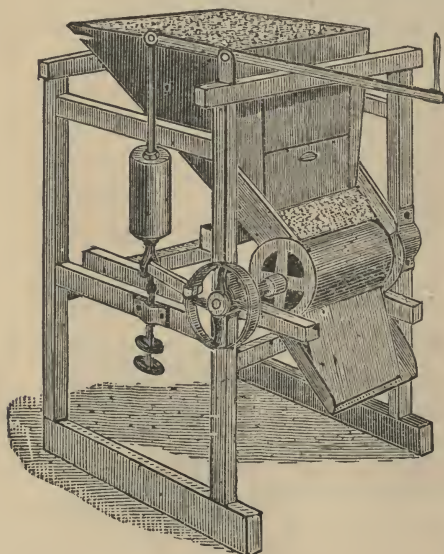
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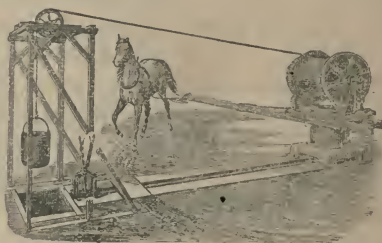
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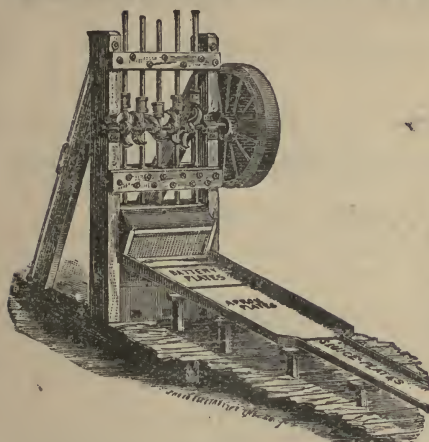
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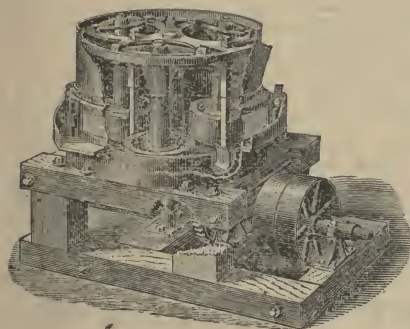
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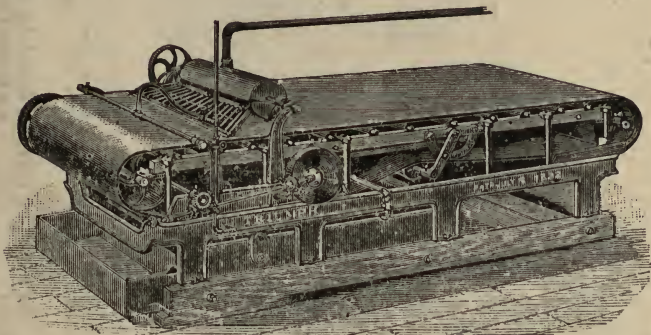
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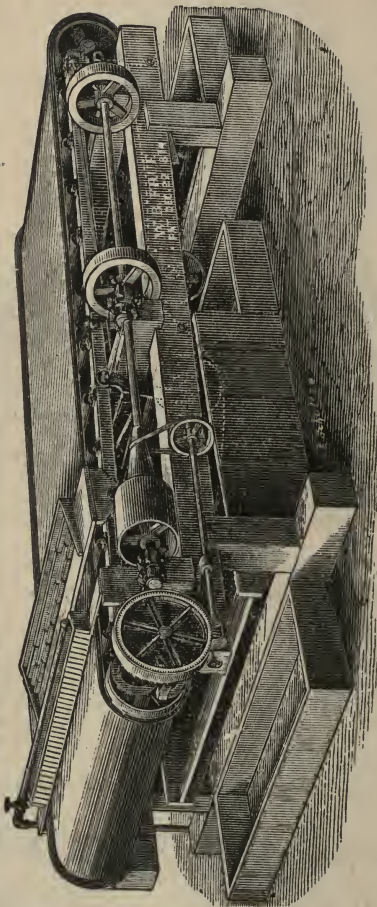
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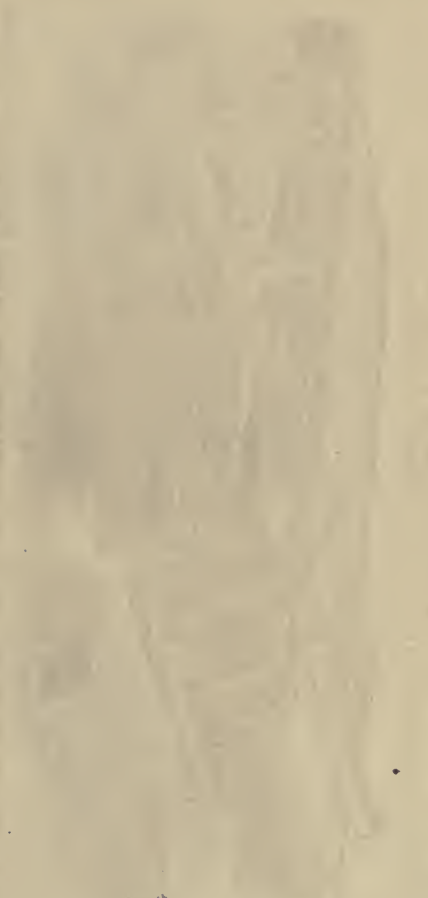
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